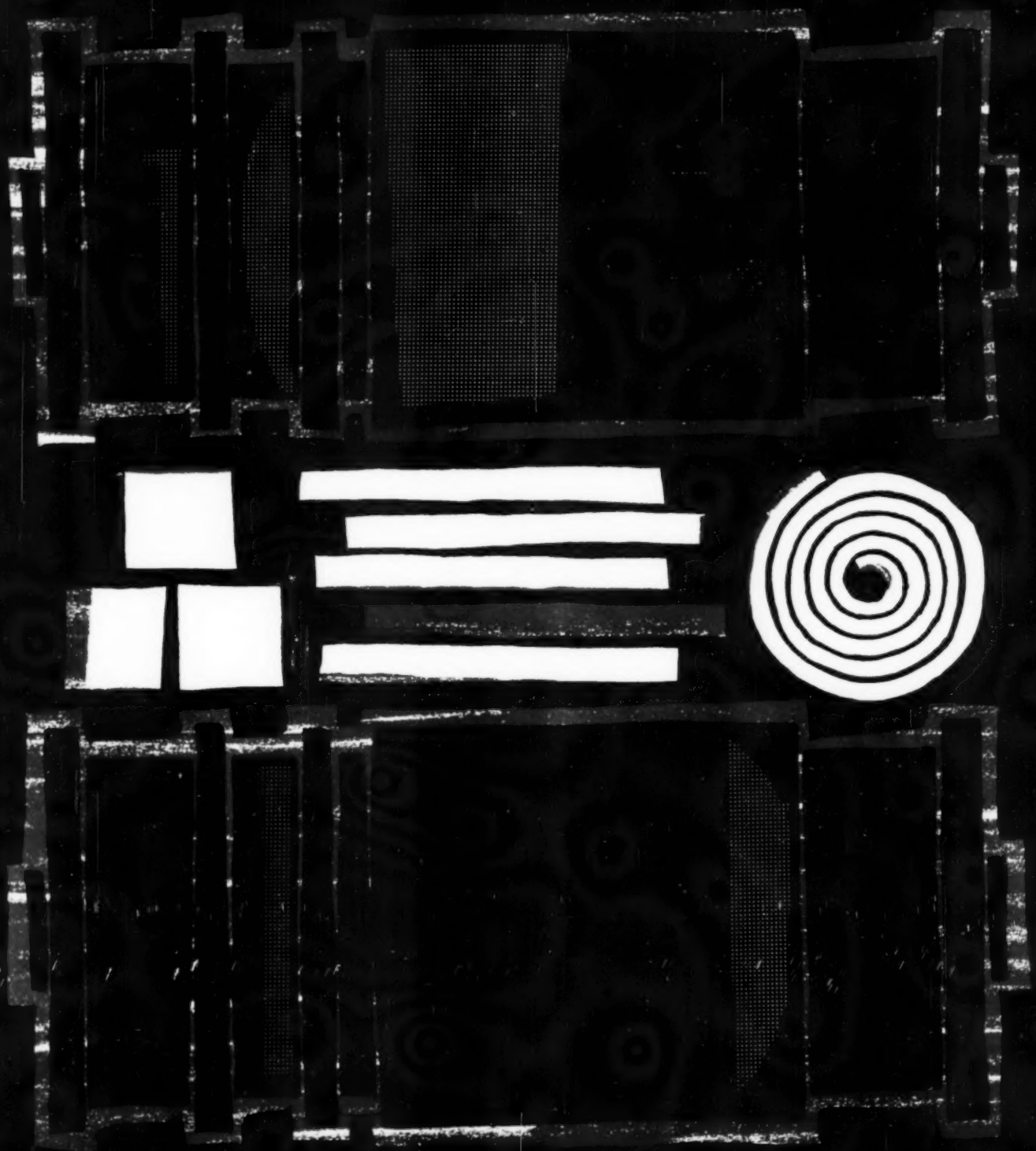
JOURNAL OF

METALS



In a quiet room at Bell Laboratories an engineer scales off the distance between two condenser microphones during a calibrating test. Able to measure air pressure variations of a few billionths of an atmosphere, such microphones play a crucial role in the scientific study of telephone instruments.

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JOURNAL OF

METALS

VOL. 6, NO. 8 AUGUST, 1954

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COVER

Taking his cue from the article beginning on p. 883, *Rare Earths Counteract Hot Rolling Defects in Stainless Steel*, artist Harry Brocke illustrates the rolling mill stage in the forming of semifinished metal products.

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Meet The Authors

H. M. Cyr (p. 900) has been associated with the New Jersey Zinc Co. (of Pa.) since 1919. He graduated from Massachusetts Institute of Technology in 1918. He is presently supervisor of metallurgical research. He is patentee of processes dealing with surface treatments of zinc oxide, manufacture of zinc oxide, and several others.

H. M. CYR

R. A. LULA

R. A. Lula (p. 883) has worked as a metallurgist abroad. In this country he has been employed by the Allegheny Ludlum Steel Corp. research laboratory since 1950. He holds degrees from the University of Bucharest and Carnegie Institute of Technology. At the present time he is research supervisor in the stainless steel section.

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C. W. SILLER

O. N. CARLSON

O. N. Carlson (p. 915) is engaged in physical metallurgy research for the Iowa State College, Ames laboratory of the Atomic Energy Commission. Born at Mitchell, S. D., Dr. Carlson graduated from Yankton College and Iowa State College. Since 1950 he has been an assistant professor of chemistry at Iowa State. As hobbies, Dr. Carlson enjoys athletics and photography.

R. H. Henke (p. 883) was born in Pittsburgh and graduated from the Pennsylvania State University in 1942, B.S. In 1943 he was with the American Manganese Steel Co., Chicago Heights. In 1944 he joined the U. S. Steel Corp. and in 1945 was employed by the A. M. Byers Co. Mr. Henke joined the Allegheny Ludlum Steel Corp. in 1945. He worked in sales development in 1947 and in 1950 was assistant chief metallurgist. In 1953 he was made manager of quality control of the Brackenridge mill. He is presently located in Pittsburgh.

R. H. HENKE

M. A. OREHOSKI

M. A. Orehoski (p. 891) has been associated with the U. S. Steel Corp. during his entire career in industry. While attending evening classes at Carnegie Institute of Technology (B.S.) he was employed at the Duquesne Works. From 1940 to 1950 he worked in the metallurgical dept. at Duquesne in the process, control, and development sections. He has held the positions of metallurgist, laboratory foreman, and chief development metallurgist, respectively, in the plant. Mr. Orehoski has been research technologist in the research and development laboratory at Pittsburgh since 1950. He co-authored two previous papers published in 1945 and 1946.

N. R. Arant (p. 891) is employed at the research and development laboratory of U. S. Steel Corp., Pittsburgh, where he is engaged in studies relating to melting and rolling. He was previously employed at the electric furnace shops of University Cyclops Steel Corp., and of Republic Steel Corp. He has also worked in the open hearth shop and the strip mill of the latter company. Born in Cleveland, he holds degrees from Case Institute of Technology and the University of Pittsburgh, (B.S., M.S.). Mr. Arant has membership in AIME, AFS, and ASM, and is a registered professional engineer. In his spare time Mr. Arant is an active Kiwanian. He also enjoys managing a Little League baseball team.

J. A. Pusateri (p. 891) is supervisor of physical metallurgy, metallurgical dept., U. S. Steel Corp., Duquesne, Pa. Mr. Pusateri is a native of Pittsburgh and received his B.S. from the University of Pittsburgh. He has worked as process engineer, development metallurgist, and heat treating metallurgist for U. S. Steel.

J. A. PUSATERI

R. M. BRICK

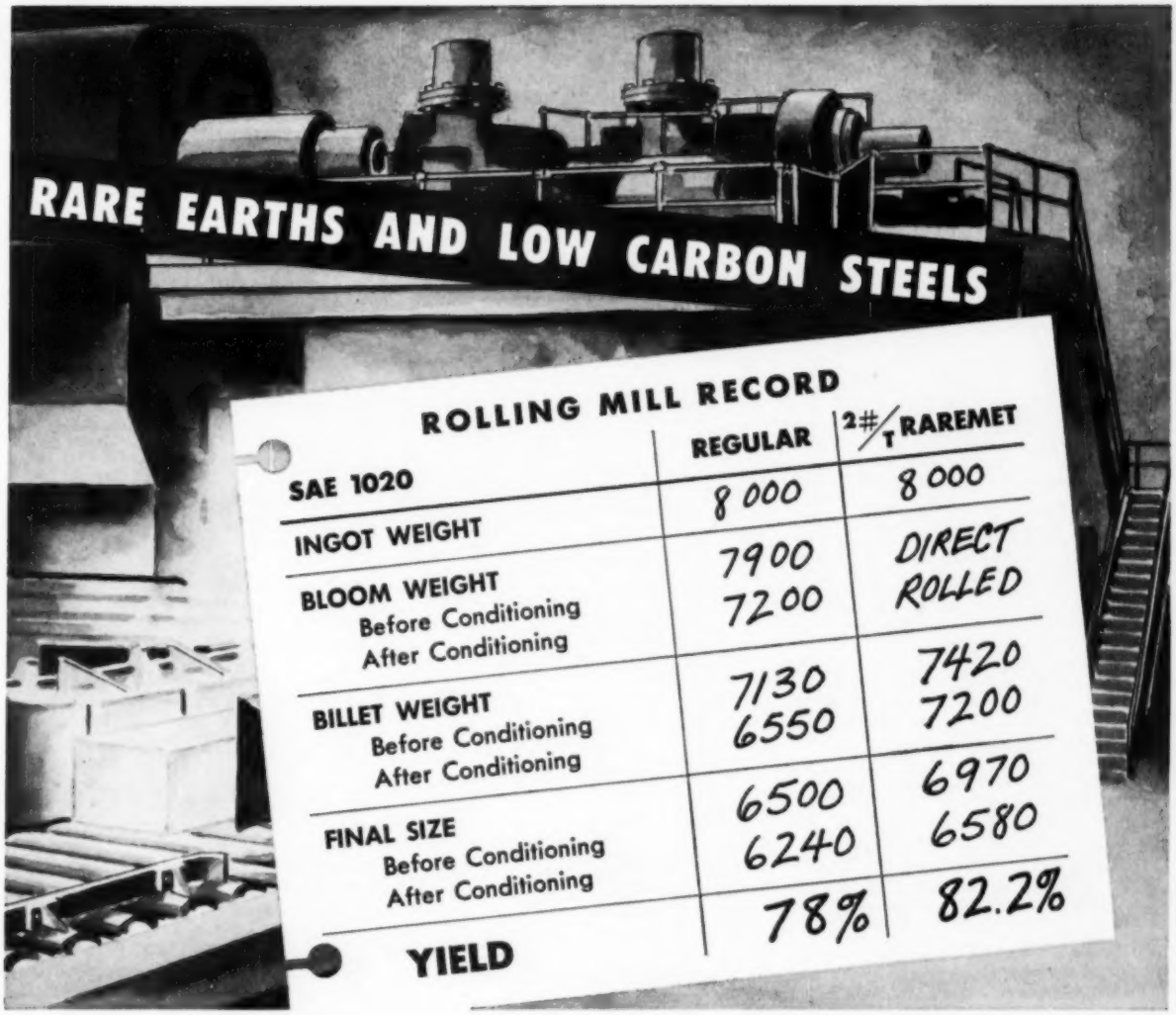
R. M. Brick (p. 906) born at Atlantic City, N. J., is a graduate of Lehigh and Yale Universities (Met.E., Ph.D.). Dr. Brick has been associated with Bethlehem Steel Co. and Aluminum Co. of America. During his time at Yale, he was a laboratory assistant and Sterling research fellow. He later became an instructor in metallurgy. From 1935 to the present he has been engaged in consulting work. He is presently with the dept. of metallurgical engineering at the University of Pennsylvania. An AIME Member, Dr. Brick was Chairman of the IMD in 1951.

B. FAZAN

S. L. FREDERICKS

Bernard Fazan (p. 919) was born in France, but graduated from the University of California, Berkeley. He attended the National School of Mines in Paris. After graduation from the university Mr. Fazan was drafted. He is presently in an armament plant in France as a trainee to become a military engineer in the reserve.

S. L. Fredericks (p. 880) is a graduate of Purdue University (B.S., 1942). Following graduation from college, Mr. Fredericks joined the Inland Steel Co., as a research chemist. He remained with this company until 1951 when he became associated with the American Steel Foundries as chief chemist of the cast armor plant. In 1953 he was promoted to his present position, chief chemist of the East St. Louis plant. Mr. Fredericks is active in the Boy Scouts.



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In addition, the Metallurgical Superintendent is responsible for the direction of a large cobalt plant, producing electrolytic cobalt from concentrate.

Initially, the engagement will be for a period of three years, but service may be extended indefinitely by mutual agreement.

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The Corporation will bear the cost of first-class transportation for the successful candidate and his family, including children up to the age of eighteen years, to Kitwe, Northern Rhodesia. In addition, an allowance will be made to cover the reasonable cost of transporting personal effects of the appointee, other than heavy furniture. On expiration of the contract, repatriation expenses will again be borne by the Corporation.

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Excellent medical facilities are provided, and recreational facilities are outstanding. The climate is temperate and healthy.

Applications, stating age, marital status, educational and professional qualifications, details of past experience and positions held should be addressed to:—

The General Manager,
Rhokana Corporation Ltd.,
P.O. Box 137,
Kitwe,
Northern Rhodesia.

Candidates are expected to be physically active and a medical certificate of health must accompany applications.

—Personnel Service—

THE following employment items are made available to AIME members on a nonprofit basis by the Engineering Societies Personnel Service Inc., operating in cooperation with the Four Founder Societies. Local offices of the Personnel Service are at 8 W. 40th St., **New York 18;** 100 Farnsworth Ave., **Detroit;** 57 Post St., **San Francisco;** 84 E. Randolph St., **Chicago 1.** Applicants should address all mail to the proper key numbers in care of the New York office and include 6c in stamps for forwarding and returning application. The applicant agrees, if placed in a position by means of the Service, to pay the placement fee listed by the Service. AIME members may secure a weekly bulletin of positions available for $3.50 a quarter, $12 a year.

POSITIONS OPEN

Mechanical or Metallurgical Engineer, young, Ph.D. in M.E., with a strong major in metallurigcal engineering, for a ballistics laboratory. Location, Maryland. W161.

METALLURGIST — B.S. in Met. Engrg. recent graduate N.Y.U., age 21, married. Educat·onal emphasis on physical metallurgy, materials engineering. Strong background in physics and chemistry. Electives: Engineering law, distribution, engineering economics, industrial management, statistical methods, quality control. Thesis: Construction of Ti-Sb phase diagram. Some business experience. Member of AIME and A.S.M. Plan graduate school in evenings. Desires production and development. Preference New York area, but not essential.

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Metallurgical Engineer, to take charge of the metallurgical problems of a mining operation. He should have a thorough knowledge of flotation, cyanidation, lixiviation and if possible, have some knowledge of running a sulphuric acid plant. Accommodations for man and wife only. Salary open. Location, Chile. F172.

Instructor for physical metallurgy. Salary, $3600 to $3800 for nine months. Location, Colorado. W131.

Foreign Sales Promotion Engineer, not over 30, graduate, who majored in nonferrous metallurgy. Will be given one year's training here in States, after which he will be permanently located in Latin America. Must be able to speak and write Spanish fluently. F125.

Metallurgist with degree. Must have had two to six years experience in the field; and have theoretical and practical experience in welding and brazing; background in high temperature alloys. Salary, $5220 to $8400 a year. Location, Maryland. W111.

Metallurgical Superintendent, 40 to 50, with extensive knowledge of copper metallurgy. Administrative experience in a senior executive capacity essential. Will do supervision and direction, through plant superintendents, of complex metallurgical operations involving the concentration, reverberatory smelting and electrolytic refining of copper on a large scale, as well as ancillary operations such as the production of sulphuric acid from converter gases; direction of a large cobalt plant, producing electrolytic cobalt concentrate. Salary commensurate with experience and qualifications, plus cost of living allowance and cash bonus. Transportation for candidate and his family paid by company to and from location. A house is available immediately, free of charge. Location, Northern Rhodesia. F105.

Metallurgical Engineer, with at least 10 years experience in design, operation and maintenance of steel mill including ore facilities, furnaces, rolling mill, etc. Must be U.S. citizen. Salary, $12,000 to $15,000 a year. Location, Far East. F92.

Junior Metallurgist with nonferrous experience for research in copper powder, electrodeposition, atomization, reduction. Salary, $4420 to $5460 a year. Location, New Jersey. Y9849.

Sales Engineer, 30 to 45, metallurgical engineering degree, with industrial sales experience. Will sell electrical contacts, thermometals, powder metals, and special purpose alloys. Salary, $5200 to $7200 a year. Location, Newark, N.J. area, and later assignment in East or Midwest. W213.

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Columbium . . Ferrocolumbium and Ferrotantalum-Columbium.

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Tungsten . . Ferrotungsten, Tungsten Powder, Calcium Tungstate, and Calcium Tungstate Nuggets.

Vanadium . . Ferrovanadium in all grades, Vanadium Oxide (Fused), Sodium Polyvanadate (Red Cake), and Ammonium Metavanadate.

Zirconium . . 12 to 15% and 35 to 40% Zirconium Alloys, and Nickel-Zirconium.

"EM" Briquets . . Silicon, Silicomanganese, Ferromanganese, Chromium, and Zirconium Briquets.

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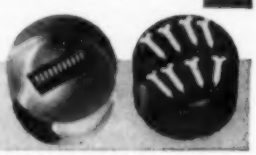

Industrial Notes.

• Buehler Ltd., manufacturers of metallurgical apparatus, has opened a new plant at 2120 Greenwood at Hartfrey Ave., Evanston, Ill. A complete display of the company's line of metallurgical sample preparation equipment, optical equipment for the laboratory, Amsler and Chevenard testing machines, and other equipment is on view at the new building.

• The new Lancaster Works of the Aluminum Co. of America, the company's first plant devoted entirely to production of fasteners and screw machine products, was formally opened. The plant also produces a small quantity of special machined magnesium parts. The new plant has 210,000 sq ft of production floor space on one level.

• *Allegheny Ludlum Steel Corp. completed expansion and improvement of its Carmet Div. plant at Ferndale, a Detroit suburb. New facilities make the company an integrated prime producer of cemented carbides for tool and die applications. For the first time the company is manufacturing all its carbide powder at its own plant. Total capacity of the division was increased 50 pct.*

• *Reynolds Metals Co. plans a $2.59 million expansion program for sheet mill facilities at Sheffield, Ala. First step is the installation of eight melting and holding furnaces in the plant's melting and alloying dept. Each of the new melting units will have a capacity of 60,000 lb, while holding furnaces will have 40,000 lb capacity. Direct chill casting units will also be installed. Four new annealing furnaces, each accommodating up to 120,000 lb of aluminum coils on trays approximately 12x40 ft will also be installed.*

• Arthur D. Little, Inc. acquired the research and development div. and laboratories of the Merrill Co., San Francisco metallurgy and engineering firm. ADL recently established a San Francisco office for its West Coast technical-economic survey work. The Merrill Laboratories, will be known as the Western Laboratories Div. of Arthur D. Little, Inc.

• A second continuous galvanizing line for production of galvanized sheet and strip is scheduled for construction at Wheeling Steel Corp.'s Martins Ferry, Ohio plant. The new installation, costing $4 million, is expected to be operating by May 1955.

• Research & Control Instruments Div., North American Philips Co., Inc., has opened a regional office at 4959 West Diversey Ave., Chicago. The new headquarters will serve Illinois, Iowa, and Missouri, and parts of Indiana, Kansas, and Nebraska.

• Average hourly payroll cost of wage earners in the iron and steel industry in April was $2.296, compared with $2.278 in March and $2.203 in April last year. Average for the first four months this year was $2.292, according to the American Iron & Steel Institute. Pensions, social security, and insurance costs, based on the 1953 average, would bring the April cost to $2.469. Total April payroll was approximately $232.64 million, compared with $249.67 million in March.

• *Aluminium Ltd. announced completion of new extensions to research facilities of its subsidiary, Aluminium Laboratories Ltd., Banbury, England. Included in the $800,000 investment are a laboratory for testing of large prototype aluminum structures for ships, bridges and other purposes; a section investigating the use of aluminum for canning; and equipment for drawing and lacquering aluminum containers and testing their behavior.*

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The Airco Carbide Injection Process in use at the American Brake Shoe Company, Mahwah, N. J.

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The injection equipment itself is simple and inexpensive. It consists of a hopper and an injection tube through which the powdered carbide is injected into the molten iron by a stream of nitrogen. The operating expenses are the cost of the gas and the carbide, trivial in comparison to the savings they make possible.

Calcium carbide injection is a fruitful result of Airco Technical Service's constant effort to find new profitable techniques for customers. If you have a welding, cutting or metallurgical problem, why not let Airco help you develop the most practical approach to it? It can pay big dividends.

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Books for Engineers

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Quality Control, second edition, by Norbert L. Enrick. *The Industrial Press,* $4.00, 181 pp., 1954—Since the first edition of this book the use of statistical quality control in industry has increased much beyond original expectations. The second edition attempts to cope with the need for addition information by the addition of Part II dealing with the following: Addition control charts for variables; control charts for defectives; control charts for acceptance inspection; and analysis of variance. While Part I of this book utilizes examples primarily from piece-parts applications, Part II introduces several typical illustrations taken from continuous and semi-continuous processing.

Ferromagnetic Domains, by K. H. Stewart. *Cambridge University Press,* $4.75, 176 pp., 1954—While the properties of ferromagnetic substances are complicated, many of them can be explained relatively simply in terms of the properties of the elementary domains into which such substances can be divided. In the last 10 years the idea of domains has been developed on a consistent basis and supported by convincing experimental evidence. After an introductory survey, this book considers in detail the various factors affecting the behavior of individual domains. It is then illustrated that by paying attention to certain effects of demagnetizing fields which have been neglected until recently, a consistent picture can be built up of the way in which the domains are combined in actual ferromagnetic substances.

X-Ray Diffraction Procedures for Polycrystalline and Amorphous Materials, by Harold P. Klug and Leroy A. Alexander. *John Wiley & Sons, Inc.,* $15.00, 716 pp., 1954—First three chapters of the book are concerned with elementary crystal geometry, the production and properties of X-rays, and the interaction of X-rays and crystals. Sufficient introduction is given so that the reader lacking previous knowledge of the topics can grasp them quickly. Some 115 pages are devoted to the first complete treatment of the Geiger counter spectrometer to appear in book form. The publishers report that the book brings together in a single volume the most useful manipulative interpretative details of the sciences of crystallography and X-ray diffraction. The book is directed at the beginner as well as the experienced researcher.

Corrosion. *Interscience Publishers, Inc.,* $1.75, 60 pp., 1953—The book is a series of papers reprinted from *Research,* a London monthly. Titles which appear in the volume are: *Metallic Corrosion,* by W. H. J. Vernon; *Prevention of Corrosion of Ships in Sea Water by Means of Cathodic Protection,* by K. N. Barnard; *Prevention of Corrosion by Metallic Coatings,* by U. R. Evans; *The Protective Action of Paints,* by J. E. O. Mayne; *Prevention of Corrosion by Means of Inhibitors,* by R. S. Thornhill; *Corrosion Testing,* by S. C. Britton, and *Vapour Phase Inhibitors,* by H. L. Bennister.

Yearbook of the American Bureau of Metal Statistics, thirty-third annual issue. *American Bureau of Metal Statistics,* $3.00, 122 pp., 1954—The book covers the year 1953. The American Bureau of Metal Statistics endeavors to present a complete statistical picture with respect to the economics of the nonferrous metals on a world-wide basis. The bureau is not connected in any way with Government but is in constant and close cooperation with Government departments. Among metals covered are copper, lead, zinc, gold, and silver. Other metals are lumped under a miscellaneous contents listing.

International Petroleum Register, thirty-first edition. *Palmer Publications,* $15.00, 708 pp., 1954—This is a yearly directory of the active oil companies of the world. It gives the history, capital structure, personnel, location, and capacity of refineries and manufacturing plants; production capacity and location; marketing facilities and areas of distribution; in fact, all pertinent data pertaining to the world's oil companies.

Metal Statistics 1954. *American Metal Market,* $3.00, 848 pp., 1954—The book contains current as well as historical records of production, consumption, stocks, imports and exports, monthly and annual market price averages, and information on miscellaneous economic subjects.

Minerals for the Chemical and Allied Industries, by Sydney J. Johnstone. *John Wiley & Sons, Inc.* $11.50, 692 pp., 1954—The author desired to present in concise form essential information on the properties of minerals and metals used in chemical and allied industries. The book covers properties, source of supply, processing and metallurgy, and uses with special reference to specifications established in particular industries. Basis for the volume first appeared as a series of articles in the *Industrial Chemist and Chemical Manufacturer.*

Adhesive Bonding of Metals, by George Epstein. *Reinhold Publishing Corp.,* $2.95, 218 pp., 1954—Object of this book is to place information in the hands of the engineer or technician which will enable him to determine if an adhesive-bonded joint will be advantageous, what type of adhesive to use, how to use the adhesive, and how to design the joint for optimum performance. Adhesives which are most generally employed with metals are considered with particular emphasis.

Residual Stresses in Metals and Metal Construction, edited by W. R. Osgood. *Reinhold Publishing Corp.,* $10.00, 363 pp., 1954—This volume was prepared under the guidance of the Committee on Residual Stresses of the National Academy of Sciences. It describes and carefully evaluates the effects of residual stresses upon the performance of various kinds of structures. Particular attention is given to the possible contributions of these structures to fracture. A wide range of information is contained in the 22 papers contributed by specialists both here and abroad. In addition, a special summary prepared by the committee covers many aspects of the residual stress problem.

Index to the Literature on Spectrochemical Analysis, Part III, 1946-1950. *American Society for Testing Materials,* $4.50, 226 pp., 1954—This is the third of a series of bibliographical surveys of the literature of spectrochemical analysis. This volume contains 1264 references. References and abstracts are listed chronologically and in alphabetical order of the first author's name in each year. An authors index and subject index are included.

Monomolecular Layers, edited by Harry Sobotka. *American Assn. for the Advancement of Science,* $4.75, 207 pp., 1954—Aim of the publishers is to present a cross-section of the recent progress in monomolecular layers. Far from attempting a comprehensive treatment, the contributors hope to assemble a symposium that will illustrate the scope and strength of their methods and techniques and will direct the reader to more detailed publications.

Forming and Bending Kaiser Aluminum. *Kaiser Aluminum & Chemical Sales, Inc.,* 260 pp., 1954—Three objectives sought by publication of this book are: Placing of comparative material on different types of forming equipment in the hands of management; the offering of engineers of specific data on aluminum characteristics which may be used from a design standpoint; and to outline in detail practical forming and bending procedures.

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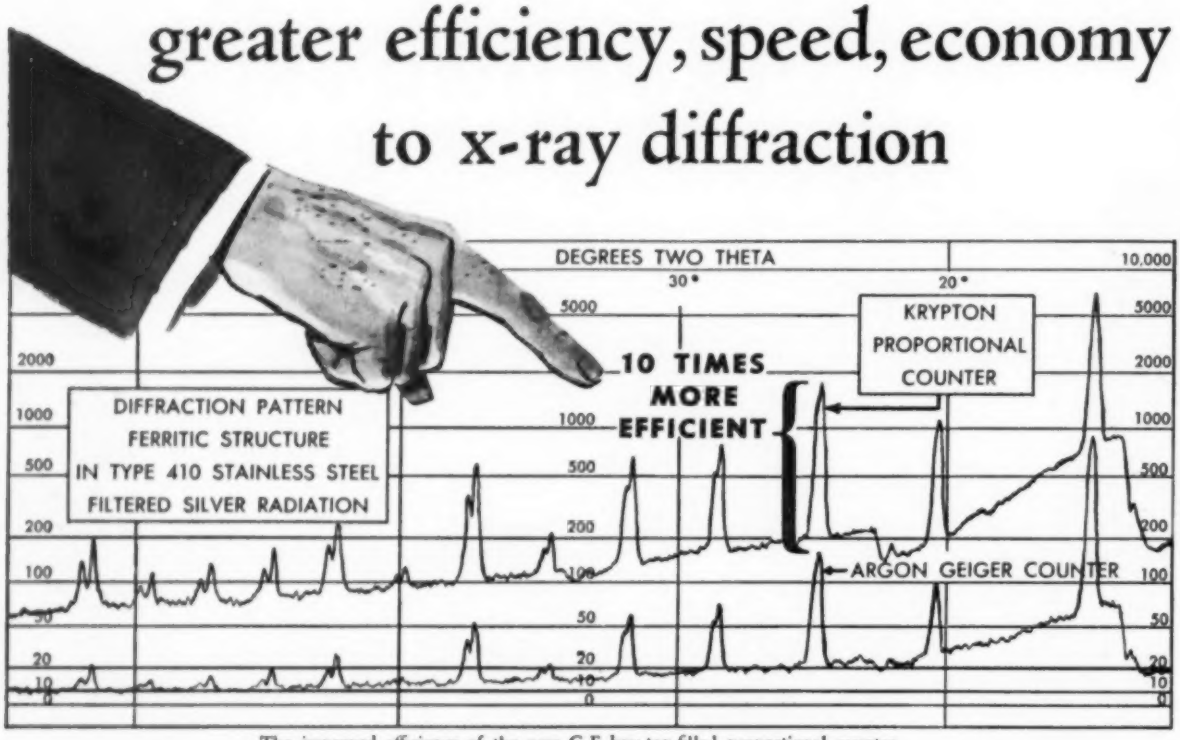

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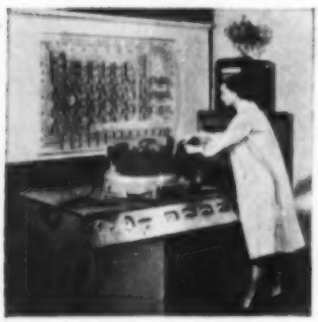

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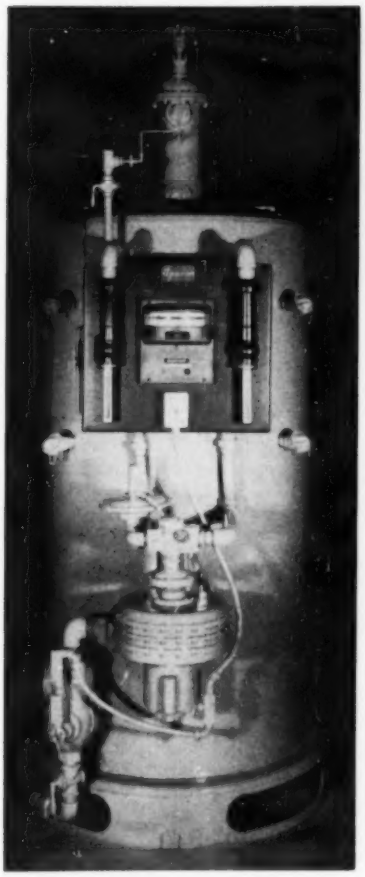


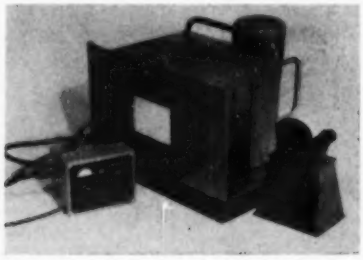
1—GAS GENERATORS: Endothermic gas generators for controlled atmosphere furnaces, producing 1250 cfh output capacity have been designed by *Ipsen Industries, Inc.*

2—UTILITY HOIST: This electrically powered hydraulic hoist permits one man to lift up 2000 lb in less than half a minute. The hydraulic system rated at 5000 psi provides power to lift 1000 lb load 8 ft. Unit has built-in boom extension. The 6-v motor is battery powered. *Unit Mfg. Co.*

3—SE RECTIFIER: An automatic selenium rectifier for heavy duty production testing is reported to have an adjustable output voltage ranging from 3 to 15 v, 25 to 120 amp dc, filtered to 1 pct rms ripple, with automatic constant voltage ±1 pct. *Richardson-Allen Corp.*

4—METAL PRIMER: A material used for priming rusted metal surfaces which are impractical to sandblast has been added to the line of protective coatings manufactured by *Emjay Maintenance Engineers.* When applied like ordinary paint directly on rusted areas, KIP 100 converts the oxide film into absorbent, inert, and stable phosphates. Paint is then applied directly over the phosphate coat, which forms an excellent bond.

5—X-RAY UNIT: A new MG 60 fluoroscopy and radiography unit for industrial inspection, production control and scientific purposes has been announced by the *North American*



Philips Co., Inc. It is extremely useful for X-ray examination of objects made from light metal, rubber, plastics, porcelain, and glass. High voltage is controlled by means of a switch in steps of 5 kilo volts peak between 30 and 60 kilo volts peak.

6—TESTING MACHINE: A portable testing machine with 200,000 lb capacity can be used in field and laboratory testing of concrete cylinders, beams, and other construction materials. No electrical or pressure connectors are required. Loads are developed by means of a hand operated, two speed concentric pump which actuates the piston of the main hydraulic system. *Soiltest Inc.*

7—PLASTIC: Protective windows in the control cockpits of open hearth furnace charging machines are made of a new high temperature transparent resin, HT-CR-39. Developed by *Cast Optics Corp.* this material is reported to have strong impact resistance and to withstand short duration exposure to temperatures as high as 350°F with no loss of clarity or rigidity. It is also claimed to be resistant to abrasion, hot metallic spatter, and all types of chemical fumes and solvents.

8—ATOM MODELS: A set of scaled atom models advances the magnification of spheres of action of single atoms to 1.5×10^8. Different colors are assigned to the various elements, which include hydrogen, carbon, oxygen, nitrogen, sulphur, phosphorus, silicon, fluorine, chlorine, bromine, and iodine. *Arthur S. LaPine & Co.*

9—BOX FURNACE: Large scale application of radiant tubes to toolroom size furnaces has been done successfully with *Lindberg Engineering Co.'s* new gas fired radiant tube atmosphere furnace. It is ideally suited for heat treating virtually all production and tool steels except high speed. Maximum operating temperature to 1850°F. New design makes it possible to suspend the tubes along the sides of the furnace chamber eliminating the need for great strength required for horizontal tubes.

10—CONTROL: Instrument built to control a vacuum distillation process is provided with eight sockets to take the plug connectors of eight resistance thermometer bulbs. The instrument measures and controls to 400°C. *Weiller Instruments Corp.*

Free Literature

20—REFRACTORIES: Technical article on current refractory practice as applied to copper smelting is available from *Harbison-Walker Refractories Co.* The use of refractories in ore melting reverberatory furnaces, converters, holding furnaces, and smelter anode furnaces is discussed.

21—COMPONENT PARTS: A catalog containing complete information on circuit breakers, disconnect switches, open knife switches, and component parts used in switchboards and panelboards is available from *General Electric Co.*, Trumbull Components Dept. More than 2500 catalog numbers are listed. It is designed specifically for original equipment manufacturers who incorporate electrical components into their products for resale.

22—ELECTRICAL CONDUCTORS: Booklet entitled *Alcoa Research and Progress in Electrical Conductors*

has been published by *Aluminum Co. of America*. Examples of aluminum conductor installations are shown. The history of Alcoa conductor research and various developments are included.

23—DUCTILE CAST IRON: A reprint on this iron, which possesses the process advantages of cast iron and has engineering properties that approach those of cast steel, is available from *International Nickel Co., Inc.* Illustrations, graphs, and charts are included.

24—HARDINGE FACILITIES: *Hardinge Mfg. Co.*, has published a folder describing plant facilities. It emphasizes the advantages of a medium-sized manufacturing firm for *farmed out* work. Illustrated are the machine shops, plate shop, Meehanite foundry, and pattern shop.

25—GAS ANALYSIS: Electro conductivity gas analyzers by Davis with instrumentation by *Minneapolis-Honeywell Regulator Co.*, are described and illustrated in new instrumentation data sheet 10.15-12. The brochure presents the theory, operation, and applications for these sensitive gas analyzers and recorders.

26—REFRACTORIES: Handbook giving details of complete line of refractories is offered by *Walsh Refractories Corp.* Research and quality control, fire clay brick, standard and series shapes, glass industry refractories, and high temperature refractory specialties are just a few of the items that are covered in the handbook.

27—VACUUM FURNACES: Applications of vacuum furnaces in melting and casting Ti, Zr, Ge, and similar rare metals are described in brochure published by *F. J. Stokes Machine Co.* Sintering, annealing, hardening, brazing, and degassing are included in bulletin 790.

28—MINERAL DRESSING: Publication of bulletin No. 20 on Cyanamid reagents has been announced by *American Cyanamid Co.* Properties and applications of various chemicals used in ore beneficiation plants and processes are described. Flotation practice for various metallic ores, and a summary of flotation methods used for treatment of various nonmetallic minerals are contained.

29—KOLDFLO: Examples of how certain tubular products are formed by this process are contained in catalog published by *Mullins Mfg. Corp.* It points out the design possibilities of this process of moving steel in its cold state.

30—GAS MEASUREMENT: A highly accurate instrument and sampling system for continuous measurement of a high purity gas stream is illustrated and described in bulletin issued by *Arnold O. Beckman, Inc.* The instrument is illustrated and a typical sampling system is presented in schematic form.

31—BE-CU STRIP: Information on a new type of mill hardened beryllium copper strip that requires no heat treatment is given in bulletin 2 available from *Penn Precision Products, Inc.* It describes the material supplied with thickness and flatness tolerances not previously available.

32—ORINOCO: The full story of the development of Orinoco Mining Co.'s Venezuelan iron ore deposit is told in book 2509 published by *Link-Belt Co.* Detailed information on the ore handling system is given, containing maps, schematic drawings and erection photographs. A photographic report shows the Puerto Ordaz installation in full scale operation.

33—TENSIONING: Catalog gives detailed information and charts on characteristic properties of Roebling wire and strand. Descriptions and photographs of various construction jobs in which prestressing has been used are included in catalog issued by *John A. Roebling's Sons Corp.*

34—GRINDING WHEELS: Catalog 1748 describes reinforced resinoid line of grinding wheels offered by *Norton Co.* Typical applications, tables of wheel sizes, prices, operating speeds and other data are shown.

35—X-RAY DIFFRACTION: Folder on X-ray diffractions is available from the *North American Philips Co., Inc.* Subjects such as stress and strain analysis, determination of preferred orientation, determination of grain size, and phase diagrams are covered.

Journal of Metals **August**
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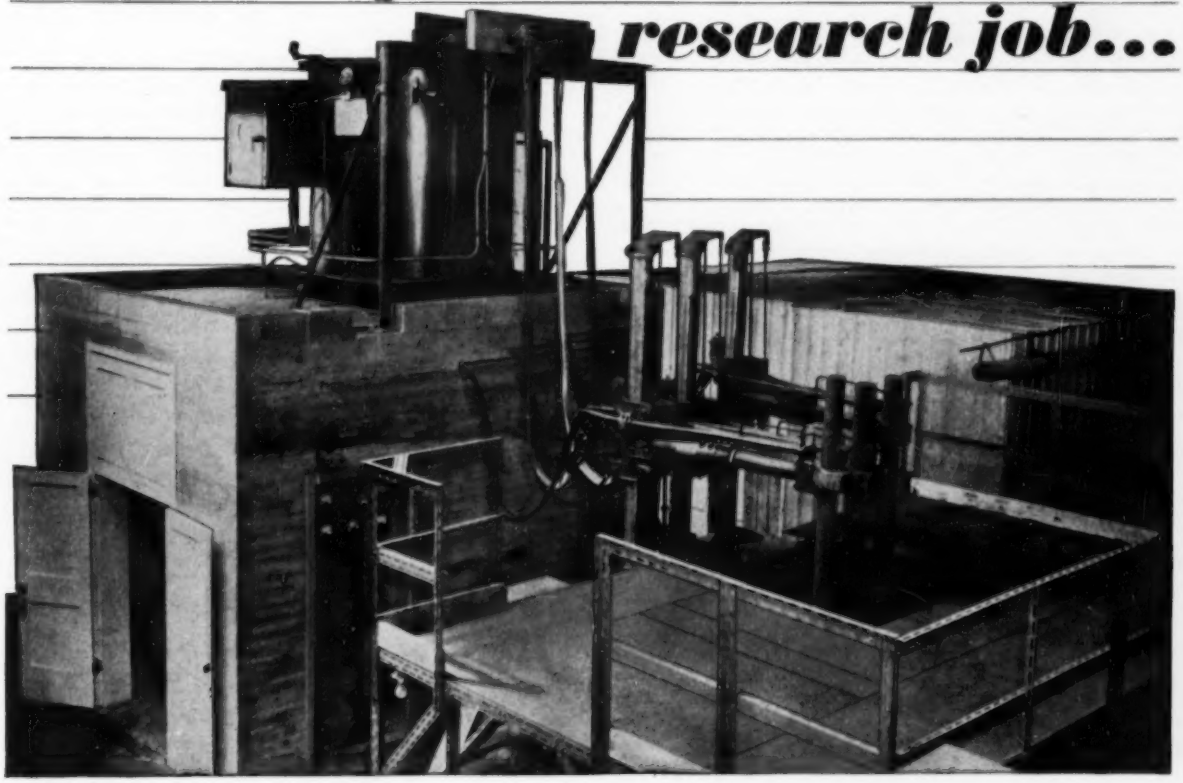
*Lectromelt** goes to Canada on a research job...



...Department of Mines & Technical Surveys installs

200KVA Lectromelt Furnace for experimentation

Cheap electric power and plentiful mineral resources have led to Canada's exploration of the economies and efficiency of electric smelting. A laboratory has been set up to test high temperature smelting and to determine whether more desirable products can be obtained more practically by using electric furnaces.

Highly satisfactory results already have been obtained in test production of ferroalloy-type material, according to the Division Chief heading the laboratory.

It is anticipated that certain ores will be smelted experimentally to obtain informative data on costs and chemical reactions encountered. Also, the smelting problems of various refractory materials will be investigated.

Expectations are that the cost of the pilot laboratory will be insignificant compared with the economies and material improvements that will result from this Canadian research. Perhaps you could benefit similarly by installing comparable research facilities.

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Journal of Metals

TWO refractory plants in Spain which have recently installed new equipment threaten to surpass the best product the rest of Europe can offer. Didier-Mersa, S. A., of Lugones (Asturias) and Aristegui Material Refractario, Ltd., of Hernani (Guipuzcoa), have spent more than $256,739 for English and German equipment. Dorsetener and Lacis presses, Eirich mixers, Lockers screens, Kennedy and Symons crushers, and other machines are described as placing the Spanish plants in a position to equal or better the quality of production from their best European competitors.

Spain consumes about 100,000 tons of refractories annually, according to *Mineral Trade Notes*, published by the U. S. Bureau of Mines. Some 5000 tons of the total is imported. Refractories consist almost wholly of silica blocks for coke and glass industries, magnesites, and chrome-magnesite bricks, and some alumina bricks for special purposes.

The nation's 20 refractory plants have a total maximum production of about 120,000 tons, but shortage of an adequate coal supply is blamed for the reduced output. Four of the factories produce more than 10,000 tons per year. About 70 pct of the plants are located in the north of Spain.

With what is termed a nominal capital investment of approximately $513,479 the Aristegui firm operates factories in Hernani, Guipuzcoa, and Vizcaya. They turn out super duty basic bricks of silica, magnesite, and chrome-magnesite. Operating name is Sociedad General de Productos Ceramicos.

Didier-Mersa, S. A. is controlled by Materiales Especiales Refractorios S. A. which is tied in with the Quijano steel family of Santander. According to an industry source, Didier-Mersa has been awarded the contract for coke ovens to be installed at the new government-sponsored steel plant in Aviles now under construction.

Spain's refractory makers are members of the Union Española de Fabricantes de Material Refractario. The group has been admitted to the Federation Europeonne des Fabricants de Produits Refractaires. One of the greatest problems of the industry in Spain is the wide variation in sizes needed by the domestic steel plants arising from failure of the Spanish organization to set up fixed commercial classification standards. Steel plants pay the same price per ton of bricks of all sizes and dimensions, just as if they were buying square or standard bricks of normal dimensions. Production costs are raised considerably because of the necessity of making many different brick sizes.

Approximate factory sale prices per ton are: silica bricks, $51; magnesite bricks, $103; and firebricks, $41. An industry spokesman analyzing the cost of silica brick figured that for every ton of production about $10 worth of raw material, $10 worth of coal, and electric power at $0.02 per kw-hr are needed. Virtually all plants, with the exception of the two already mentioned, are only partly mechanized requiring much hand labor. The average handmolder works about 280 eight-hr days per year, earning approximately $513.00. Spanish tariff on imported brick is roughly $14.00 per ton.

STANDING on steel legs rooted in bedrock and extending 132 ft below street level, San Francisco's first skyscraper is starting to take shape under the rigid earthquake code adopted in 1948. When the code was passed there was a widespread belief that the restrictions it imposed would bring an end to skyscraper construction in San Francisco. The building, under construction for Equitable Life Assurance Society, will be San Francisco's first tall building with steel foundations resting in bedrock.

U. S. Steel Corp.'s Consolidated Western Steel div. is erecting the structural framework for the 25-story building. U. S. Steel is also supplying 5300 tons of fabricated steel, 3300 tons of steel piling. and 50 tons of stainless steel.

Design of the building involves use of uniformly tapered columns and butterfly-shaped spandrel beams in the exterior walls up through the 14th floor in place of conventional straight columns and beams. Another innovation is the use of high strength steel bolting instead of rivets. While not generally associated with building construction, the immediate advantage of bolting will be to cut construction noise. Air impact wrenches make less racket than riveting hammers. Welding will also be employed in certain areas. Stainless steel will be used extensively for the exterior finish of the building and in the main lobby. Facade will combine steel, aluminum, and white marble.

Another piece of building history was made recently on an early summer day in New York City. Starting at about 6:02 am, some 40 construction men swarmed over the bare framework of a new building at 57th Street and Park Ave. By 4 pm they had completely emplaced the aluminum outer curtain of the structure. Previous record, set by the same firm, Tishman Realty & Construction Co., Inc., was six and a half days. Aluminum panels were fabricated from Alcoa sheet by General Bronze Corp., who also furnished the crew of 40 men. Special supervisory personnel were also detailed to the job. Commented one construction man, "I never saw so many foremen on one job in my life."

Panels were distributed in strategic spots throughout the building. Actually, only one small detail was omitted from the master plan. The whistle that started work had to be borrowed from a passing policeman.

◆◆◆◆◆◆

THERE seemed to be a general agreement on at least one point during the recent meeting of the American Society for Engineering Education at the University of Illinois. Somewhere along the line engineering students were not being adequately trained for the job of learning to be engineers. Among the long list of influences which come to bear during the training period, L. E. Grinter, president of ASEE, trained his sights on the public school system and the employing group that accepts the product resulting from the total education cycle.

"One of the major factors that necessarily influence the character of engineering education is the

quality and quantity of secondary school preparation," Mr. Grinter said. He was of the opinion that the student's capacity to pass mathematical and scientific subject matter is indicative of the job being done by the schools, and he felt that, "a continuing loss [in that area] certainly is taking place." Mr. Grinter stated that the current attitude in public schools is one that discounts the importance of rigorous courses such as mathematics. This, despite the insistence by employers that the ability to write clearly and to make simple mathematical calculations are the most important tools high school graduates can bring to industry.

He argued against the concept that, "We can, and therefore, we must try to accomplish everything for every student." Mr. Grinter decried the system of electives that allows only a small degree of free choice. He desires a return to a system that recognizes the existence of the individual. Electives should be chosen with proper guidance, however.

He labeled the greatest weakness in engineering education as, "Neither the neglect of practice nor of specialized science but insufficient attention to welding these together so that the student can explain the rules of engineering art or practice from his scientific knowledge."

Taking a slightly different tack, Major Lennox R. Lohr, president, Museum of Science and Industry, Chicago, felt that, "More young men and women should be inspired to take technical courses. Emphasis must be placed on the basic sciences in the lower grades, and teachers must develop the technique of making them enjoyable and practical." He warned that we are losing technical advantage over the USSR where half the curriculum in secondary schools is given to science and technology and where one third of college enrollment consists of student engineers."

The report of the ASEE Manpower Committee indicated that demand for engineering graduates has fallen about 20 pct. However, because of the relatively small graduating classes there is still substantial unfilled demand.

◆◆◆◆◆◆

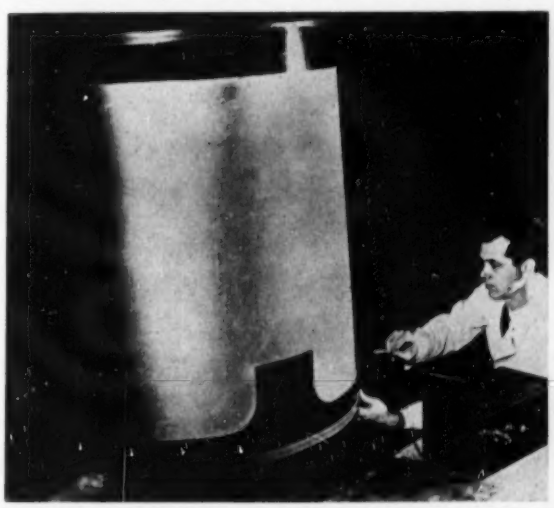
METALLURGY is saving American railroads millions of dollars every year. Research and experimentation in cooperation with the metals industry on problems arising from rail, rolling stock, bridges, and other fixed installations has served to develop new answers to long standing problems.

One of the top items in the program sponsored by the American Assn. of Railroads is rail improvement. About 1 million tons of rail are bought every year and laid as replacements. The program calls for extensive studies to redesign rail sections for more resistance to shelling and breakage. One new design effected a 25 pct increase in strength with only a minute addition of alloy. Another project which has paid off is transverse fissure research. A saving of $80 million is reported on the 16 million tons of rail made under the controlled cooling process. Controlled cooling effected elimination of shatter cracks and transverse fissures, in addition to reducing the number of vertical and horizontal split heads in all rail manufactured by the process. Rail life has been extended some 10 pct.

Another AAR project is directed at design and metallurgical improvement of railroad crossings. Type of metal used in the experiments is the regular Hadfield manganese steel, suitably heat treated. Some crossings have been produced of nickel manganese but costs have not been justified. Proving ground for the crossing was at McCook, Ill., near Chicago. Here, the double track of the Santa Fe R.R. crosses the double track line of the Baltimore & Ohio Chicago Terminal. Slow speed heavy traffic on the Terminal and high speed heavy traffic on the Santa Fe use the crossing. Various companies were invited to submit frog designs for inclusion in the test. Critical areas in the frogs were determined by use of stress coat brittle lacquer. Strain gages were then applied to those areas and stress determined under traffic. Each design remained in service until failure developed. In every instance failure occurred in the flangeways. Changes in design resulted from the measured stresses. In comparison to earlier designs giving less than one year's service, current models offer life of from five to six years.

There are some 170,000 railroad bridges in the U. S. It has been estimated that carriers redrive about 2 million rivets annually, in addition to placing 800,000 in new construction. Research determined that a new high-strength bolt can be used in place of rivets at a saving of 20¢ per bolt. The bolts offer a superior fastening. Tests also disclosed that cold-driven rivets were inferior to hot-driven rivets or bolts under repeated loading.

Most of the 55 research projects were started in the 1940's or earlier. Total savings for the life of the projects are estimated to be $1 billion with savings continuing at a rate of $100 million annually.



North American Aviation and Turco Products Inc. have developed a chemical milling process which offers contouring without use of physical force. Chem Milling can be used to produce precision contouring of aluminum forgings or complicated shaped curved sheets.

Drift of Things

FRANCE celebrated the 100 year anniversary of the industrial production of aluminum June 14 to 19. Under the sponsorship of the President of the French Republic, the Aluminum Centennial Congress offered a varied scientific and cultural program which included scientific papers, visits to plants, and an exhibition on the Seine embankment in Paris.

In this country, an American Committee was organized to cooperate with the French in bringing the event to the attention of U. S. industry. We were invited to a conference arranged by W. P. Bittenbender, chairman of the American Committee, to meet Jean Matter, president of L'Aluminium Francais, who is making a goodwill tour of the U. S. industry.

M. Matter stated that France has reached its peak in aluminum production because of the limited supply of cheap electric power. The French Alps area is the most highly developed electrical power region in the world, and no further economic sites are available. On the other hand, France has a big potential for aluminum production in its colonies. In this direction, the first plant will soon be opened in the French Cameroun in Equatorial Africa.

In commenting on the history of aluminum, M. Matter brought out the interesting fact that Emperor Napoleon III was the patron of its industrial development. Shortly after Henri Sainte Claire Deville announced his method of aluminum production to the French academy, he presented a medal of aluminum to the emperor. Napoleon III, dreaming perhaps of marching in the footsteps of his grandfather, was fascinated with the possibilities of using this new light metal as armor for the French Cavalry and authorized that experiments be continued at royal expense.

M. Matter provided us with many interesting facts about aluminum, but of all the incidents he related the one which still lingers with us concerns the first article ever fabricated from the first industrial bar of aluminum. It was not, as might be expected, an article of war or a gadget to revolutionize an aspect of industry. But, it was an article of undisguised joy and merriment . . . a baby rattle for the infant Prince Imperial! (E.M.W.)

FROM time to time on these pages we have given voice to the whys and wherefores of the reported shortage of engineers. Blame for the shortage has been placed on almost every pair of broad shoulders available—industry, universities, high schools, parents, engineers, students, government, etc. Some people have gone so far as to maintain that there is no shortage of engineers, but rather short sightedness on how available engineers are utilized.

Now along comes a report prepared by the Professional Engineers Conference Board for Industry (in cooperation with The National Society of Professional Engineers) that points up a basic problem of current interest to engineers and industry. We feel that their story *How to Attract and Hold Engineering Talent* is one that should receive wide attention, although this is not intended as an endorsement of all the findings of this group. The booklet is available from The Professional Engineers Conference Board for Industry, 1121 15th St., N.W., Washington 5, D.C. for $2 per copy, $1 to members of NSPE.

The report, the third of a series of Executive Research Surveys begun in 1951, represents a questionnaire survey of 200 companies employing engineers, and 1400 individual engineers.

Although 88 pct of the executives responding to the questionnaire felt that the engineering schools were producing graduates with an adequate technical background, 62 pct felt that these newcomers to industry were sorely deficient in other areas of study. This fact was recognized by the young engineers, and it was generally agreed that sufficient training was lacking in English, the social sciences, and business administration. There was the general feeling that industry could profit by spending more time with the engineering colleges to help construct and maintain courses that come closer to a realistic approach to industry's needs.

One of the conclusions reached was: "It (industry) could at small cost, raise morale of its engineering employees, and stimulate their professional growth and their contentment by encouragement of *prestige* activities such as participation in professional organizations and community affairs, writing for publication, and lecturing or teaching. These activities also could be utilized as a part of the company's public relations program with benefit to all concerned."

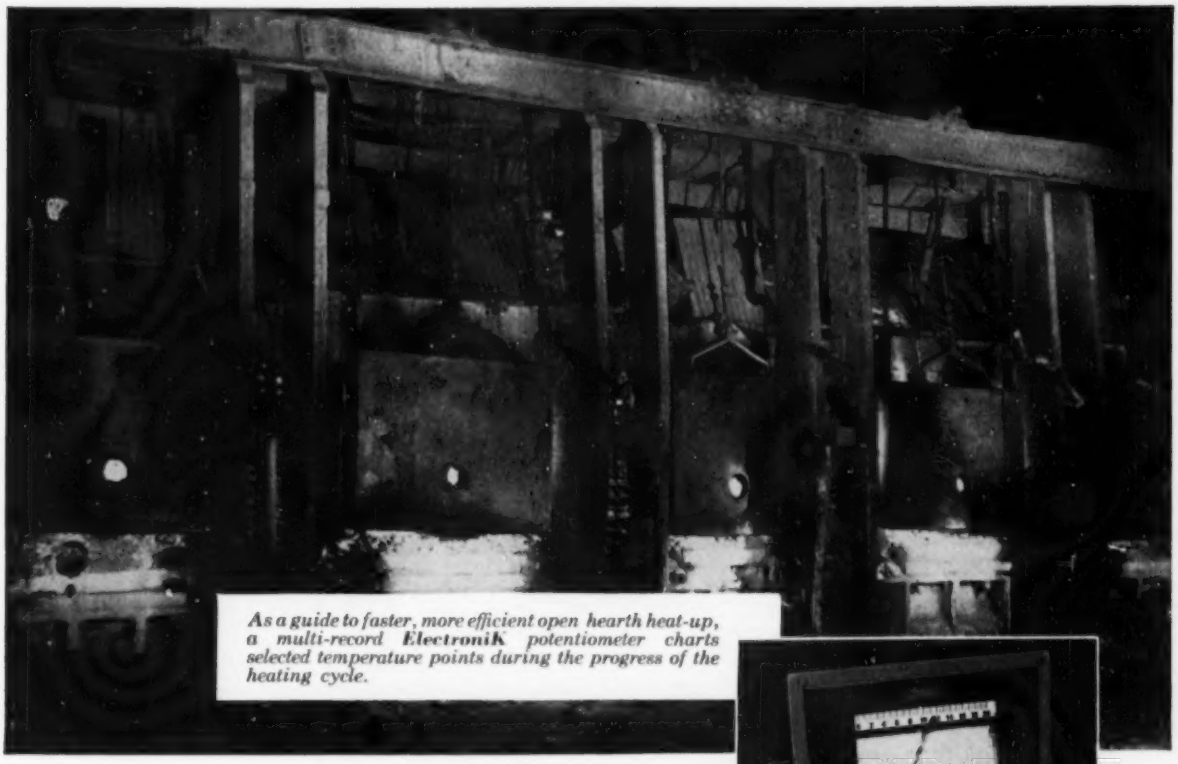
One of the most interesting parts of the report was the section dealing with unionization of engineers. The engineers answering the questionnaire indicated clearly that they wished to be considered and recognized as professional employees and wanted to maintain their individual independence. However, 34 pct of them believed that engineers' interests were strengthened economically or otherwise by membership in a collective bargaining organization. This is a substantial increase on the 5 pct figure obtained in a similar survey five years ago. Only 3 pct indicated that they actually did belong to a union.

Some 66 pct of the engineers indicated that they favored nonbargaining organizations for the purpose of bringing engineers closer to management by having some medium for the exchange of ideas and for improving communications. Many of those answering did point out, however, that such an organization frequently is the first step toward outright unionization.

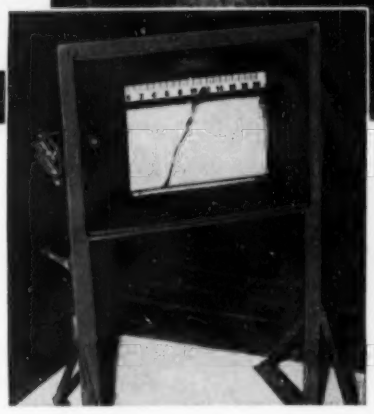
Other conclusions pointed up in the report were the readjustment of salary scales to provide more equitable compensation for the engineer who has been in the industry for a number of years, a new approach to campus recruiting to provide a more realistic presentation of the industrial facts of life to the job candidate, and differentiation in personnel policies as they apply to engineers and nonprofessional employees.

HERE'S a technical definition that you can add to your latest handbook: Management Consultant —A fellow who's smart enough to tell you how to run your business but too smart to have one of his own.

A. S. Cohan



As a guide to faster, more efficient open hearth heat-up, a multi-record ***ElectroniK*** *potentiometer charts selected temperature points during the progress of the heating cycle.*



Open hearth heat-ups speeded...costs reduced... with *ElectroniK* instruments

WHENEVER an open hearth furnace is being brought back in production after repairs, the time and expense of heat-up can be substantially reduced with the help of *ElectroniK* instrumentation. These instruments provide operators with accurate data on which to base fuel adjustments . . . makes possible thorough, even heating that adds to the life of refractories . . . permits rate of heating to be held at the fastest safe rate . . . helps keep total time and fuel consumption during heat-up to a minimum.

Two alternate instrument systems are available. Where manual regulation is preferred, an *ElectroniK* four-record strip chart instrument (shown above) records temperatures of thermocouples in the furnace at selected locations above doors and in the roof. A special chart makes it easy to compare the heating curves with the desired time-temperature relationship, to facilitate manual adjustment of fuel. For fully automatic control, an *ElectroniK* circular chart program controller regulates fuel input to make temperature rise evenly at a prescribed number of degrees per hour.

Your nearby Honeywell sales engineer will be glad to discuss the system best suited to your own shop . . . and he's as near as your phone.

MINNEAPOLIS-HONEYWELL REGULATOR CO., *Industrial Division*, Wayne and Windrim Avenues, Philadelphia 44, Pa.

● **REFERENCE DATA: Write for Data Sheet No. 6.4-11, "Faster Heat-Up for High Temperature Furnaces."**

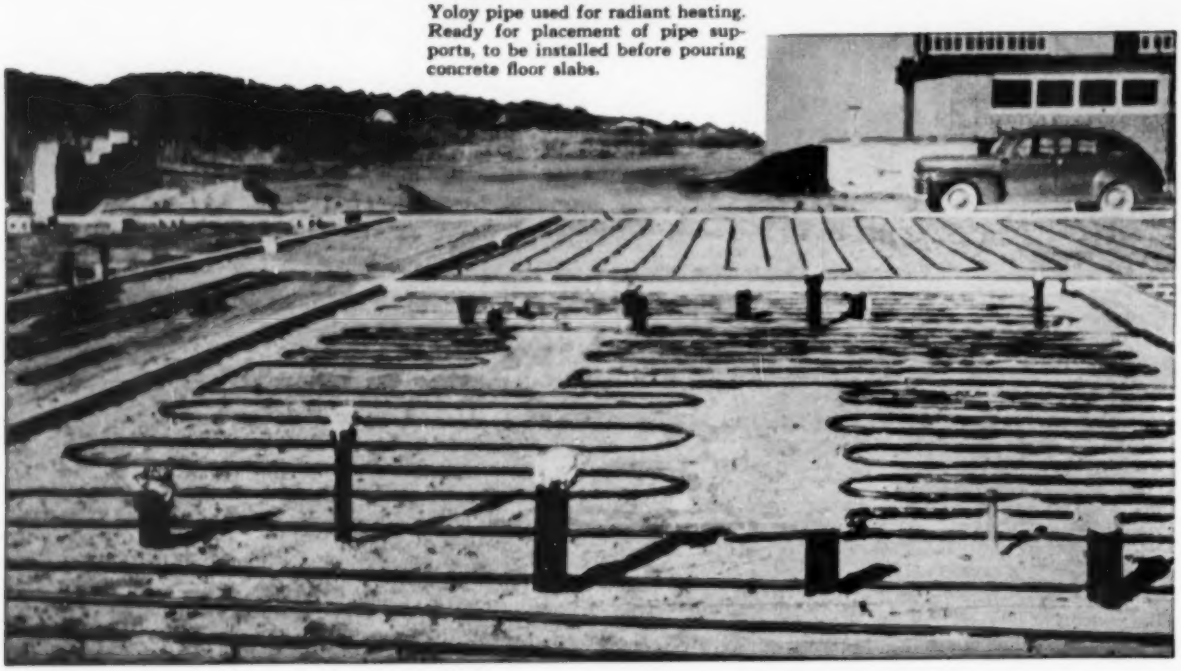
MINNEAPOLIS **Honeywell**

BROWN INSTRUMENTS

First in Controls

Where It Can Save You Money

Yoloy pipe used for radiant heating. Ready for placement of pipe supports, to be installed before pouring concrete floor slabs.



long life underground too

. . . Piping of high strength low alloy steel containing nickel provides multiple advantages

INDUSTRY has widely adopted concealed piping for such diverse applications as radiant heating indoors and removal of snow from city streets.

Regardless of its purpose, however, where buried or concealed piping is involved *only long-lasting pipe can be economical.*

You can see such piping, above, made from the same composition of nickel alloyed steel which has proven so successful in the oil, mining, railroad, chemical, trucking and other industries.

Weldable, and of high mechanical strength, it is produced by The Youngstown Sheet and Tube Company, Youngstown, Ohio, under the tradename Yoloy Continuous Weld Pipe.

In standard tests, Yoloy shows four to six times greater resistance to atmospheric corrosion than does carbon steel. Furthermore, Yoloy piping resists attack from highly sulphurous atmosphere, brine and many other corrosives.

In addition, it resists abrasion and shock to an extent unmatched by any carbon steel of equal strength.

Another valuable advantage . . .Yoloy pipe can be electric or gas welded, readily. And the welds show approximately the same strength and ductility as the parent metal.

Easy to thread and fabricate with standard pipe tools, Yoloy Continuous Weld Pipe is one more example of the improved performance and ready response to fabrication derived from steels containing nickel.

In all sorts of applications, nickel alloyed steels prove to be lowest in ultimate cost. For actually, the many standard grades available make it easy to select exactly the right one to meet any reasonable combination of fabricating and service demands.

Whatever your industry, send us details of your metal problems. We'll be glad to help you with suggestions based on wide practical experience.

EMBLEM OF INCO SERVICE

THE INTERNATIONAL NICKEL COMPANY, INC. 67 WALL STREET NEW YORK 5, N.Y.

Constructed in cooperation with Kaiser Aluminum & Chemical Corp., the largest die casting machine ever built was unveiled at the Toledo plant of the Doehler-Jarvis div. of National Lead Co. Primarily designed for aluminum the machine can turn out castings up to 75 lb. When adapted for zinc castings, the machine is expected to be able to produce castings up to 200 lb.

If the Talgo train gains a marked degree of American acceptance, it may prove the beginning of a huge new aluminum market. Built by A. C. F. Industries, Inc. in 1948, the train was recently tested by the New York, New Haven & Hartford Railroad. It is made almost entirely of aluminum except for wheels, axles, and diesel motor. The train can do better than 100 mph and takes curves at 87 mph.

It is reported that Republic Steel Corp. has shown interest in expanding its melting and fabricating facilities to handle production of titanium sponge. Talks between the corporation and General Services Administration are said to be in progress.

One of the world's biggest plants for the manufacture of titanium sponge is being planned by Electro Metallurgical Co., a division of Union Carbide & Carbon Corp. Plans call for construction of the $30 million unit at Ashtabula, Ohio. It will have a 10,000-ton annual capacity.

Bill HR 7709, which continues through June 1955 the suspension of import tariffs on copper has been signed by President Eisenhower. The bill, now known as Public Law No. 452, provides for automatic reactivation of a 2¢ per lb tax if the market price drops below 24¢ per lb for any full calendar month.

General Cable Corp. plans to manufacture for the first time in the U. S. direct-extruded aluminum sheath cables. The company has arranged to purchase an aluminum sheathing press of a type which has been successfully used in extruding aluminum sheathing directly onto an insulated cable core. The press is being purchased in West Germany.

Great Britain expects to produce 18.5 million tons of steel during 1954, according to the British Iron & Steel Federation. During the first six months of the year production is reported to have been 9.5 million tons. Last year British steelmakers turned out 17.6 million tons.

Union Miniere du Haut-Katanga has developed a process for recovery of germanium oxide from gas oven operations at Lumbumbashi. The germanium oxide is pure enough for electronic use. The powder will be enriched in an African plant now under construction.

European Meeting Beckons AIME Metallurgists In Summer 1955

IT may be too early to start packing, but it's not too soon to start thinking about attending the meetings of the Joint Metallurgical Societies in Europe during June 1955.

London, Paris, and Dusseldorf will serve as backdrops for technical sessions and jumping off points for plant tours covering some of the most significant metallurgical facilities in the resurgent European Economy. Six European metallurgical societies have extended the invitation to AIME members to participate. Some 250 conferees from the U. S. are expected to attend the three week meeting.

Plans are for the entire U. S. delegation to meet with European specialists in each of the countries visited. Rather than accent technical paper presentation, emphasis will be on formal and informal discussion of mutual problems. Many of the sessions will take place at plants where visitors will be able to get the feel of problems in actual context. Plant guides will be men familiar with technical problems.

The first draft of the program is subject to change during the months preceding the meeting. However, it offers a sound idea of what delegates can expect. Additional details of the meeting will be forwarded from AIME headquarters to those who indicate interest in attending.

While some of the plant tours and technical sessions may be closed to wives of members, the distaff side can look forward to a program which will offer them a magnificent opportunity to dip into the glamorous and cultural side of the continent. Tours covering cultural and historical aspects of Europe have been arranged—and not the least of the attractions will be journeys into the world of Paris fashions.

Thos. Cook & Son, Inc. are the official travel agents for the meeting. Delegates are of course free to make other arrangements. Round trip fare rates to Europe forwarded to AIME headquarters by Cook are: first class ship passage, $975; Cabin class, $500; Plane, first class, $738; and tourist class, plane, $540.

Cost of the basic tour in Europe is about $400 for hotel, meals, and travel. An additional charge of $120 can be anticipated by those who desire better hotel accommodations and first class railroad cars. An even higher additional cost will be tacked on to the bill of those who prefer the deluxe hotels.

If you reach Europe before the meeting starts or decide to stay for a time afterward, expenses will be higher of course. It has been estimated that the entire trip can be made from New York for approximately $1500.

Information can be obtained from the Secretary of the Metals Branch, AIME. It is requested that those planning to attend submit their names now.

Tentative Program

Wednesday, June 1, London

Registration in the morning
Opening plenary session in the afternoon
Reception and conversations in the evening

Thursday, June 2, London

Meetings of the discussion groups in the morning and afternoon
Official dinner in the evening

Friday, June 3, London

Meetings of the discussion groups in the morning
Technical visits in the afternoon
- British Iron & Steel Research Assn. Laboratories
- British Non-Ferrous Metals Research Assn. Laboratories
- High Duty Alloys and Fulmer Research Laboratories, Slough

Saturday, June 4, Near London

Technical Visits
- Oxford Laboratories and Colleges

Sunday, June 5

Departure on Sunday or before to Provinces for some technical groups, optional cultural trips for other groups

Monday, June 6, Near London

Technical Visits
- Iron and steel works of Messrs. Stewarts & Lloyds Ltd. including basic Bessemer and electric furnaces and tube works, Corby
- Enfield Rolling Mills Ltd. including copper and brass smelting and wire rod mills

Tuesday, June 7, Near London

Technical Visits
- Cambridge University, laboratories and colleges
- National Physical Laboratory, Teddington

Monday, June 6, Swansea

Technical Visits
- Steel Co. of Wales Ltd., Margan and Abbey Works
- Mond Nickel Co. Ltd., Clydach
- Aluminum Wire & Cable Co. Ltd.
- I. C. I. Ltd., metals div., Waunarlwydd

Tuesday, June 7, Swansea

Technical visits
- Trostre Works of Steel Co. of Wales
- Works of Guest Keen Baldwins Iron & Steel Co. Ltd., and Guest Keen & Nettlefolds Ltd. at Cardiff
- Rogerstone Works of Northern Aluminum Co. Ltd.

Monday and Tuesday, June 6 and 7, Birmingham

Technical visits
- British Aluminium Casting Co. Ltd.
- Mond Nickel Research Laboratories and Henry Wiggin & Co. Ltd.
- James Booth & Co. Ltd.
- I. C. I. Metals Div., Witton
- Stewarts & Lloyds Ltd., Bilston
- Round Oak Steel Works Ltd., Brierley Hill
- Walter Somers Ltd., Halesowen
- The University of Birmingham

Monday and Tuesday, June 6 and 7, Sheffield

Technical visits
- English Steel Corp. Ltd.
- Thos. Firth & John Brown Ltd.
- Hadfields Ltd.
- Samuel Fox & Co. Ltd.
- British Iron & Steel Research Assn., laboratories
- United Steel Companies, Ltd., central research laboratories
- The University of Sheffield

Monday, June 6, Scunthorpe

Technical visit
- Works of Appleby-Frodingham Steel Co. (branch of the United Steel Companies Ltd.)

Tuesday, June 7, Scunthorpe

Technical visit
- Normanby Park Steelworks of John Lysaght's Scunthorpe Works Ltd.

Monday, June 6, North-East Coast

Technical visit
- Works of Consett Iron Co., Ltd.

Tuesday, June 7, North-East Coast

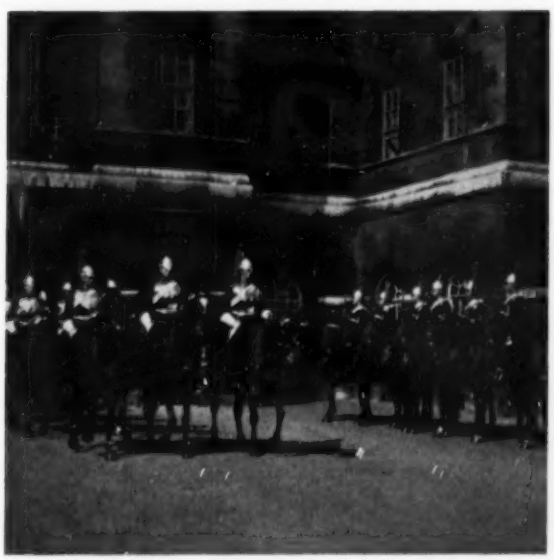
Technical visits
- Dorman Long & Co. Ltd. (Lackenby Works)
- Head, Wrightson & Co. Ltd.
- Ashmore, Benson, Pease & Co. Ltd.

Monday, June 6, Scotland

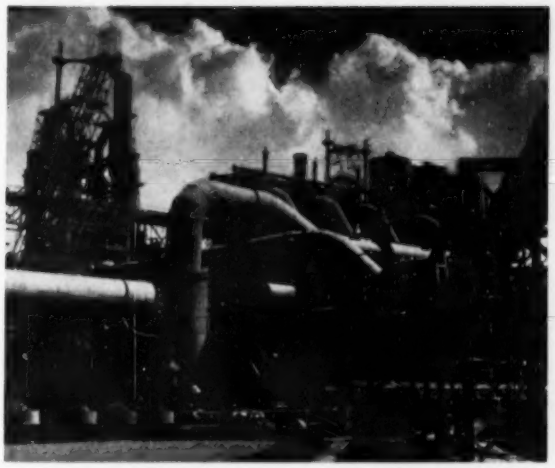
Technical visits
- Clydebridge Steel Ltd. and Clyde Iron Works of Colvilles Ltd.
- Works at Thornliebank of Henry Wiggin & Co. Ltd.
- Clyde Alloy Steel Co. Ltd.
- British Aluminium Co. Ltd. Falkirk

Wednesday, June 8

Travel from London to Düsseldorf



One of London's most impressive ceremonies is the changing of the guard at Whitehall.



Huttenwerk Oberhausen A. G. is located about 12 miles north of Dusseldorf. The plant is an important one to West German steel production.

Thursday, June 9, Düsseldorf

Registration and Opening Session in the Eisenhuttenhaus in the morning. Welcome by Professor Dr. H. Schenck (steel) and Professor Dr. P. Brenner (nonferrous metals)

Technical visits in the afternoon
- Mannesmann-Hüttenwerke A. G., Duisburg-Huckingen
- Hüttenwerk Rheinhausen A. G., Rheinhausen
- Hüttenwerk Oberhausen A. G., Oberhausen
- Duisburger Kupferhütte, Duisburg
- Max-Planck-Institut, Düsseldorf

Reception by the Council of the City of Düsseldorf in the evening

Friday, June 10, Düsseldorf

Meeting of the Discussion Groups in the morning
- Blast-Furnaces and Coke-Oven Practice. Discussion Leader: Dipl. Ing. H. Kahlhofer
  - Burden Preparation (crushing, classifying, sintering, pelletising, low-shaft furnace)
  - Coke, Properties and Testing (reactivity of coke and its behaviour in the blast furnace)
- Steelworks Problems. Discussion Leader: Dr. A. Schildkotter
  - Methods of increasing the rate of refining (pre-refining of pig iron with oxygen, blowing with oxygen and with oxygen-enriched blast)
  - Improvement of the life of open hearth furnaces (use of new type silica brick, all-basic roof)
  - Temperature measurement and control in steelworks operation with particular reference to the measurement of the temperature of steel heats with the optical pyrometer in order to improve the quality and reduce the scrap
- Rolling Mill Problems. Discussion Leader: Dipl. Ing. E. Wilms
  - Application of rectifiers for reserve drives for mill trains
  - Neutralisation and use of pickling waste (in conjunction with a visit to the Bochumer Verein)

Technical visits in the afternoon
- Brown-coal (lignite) mine with power station; also to the allweg railway in the Cologne area
- Works of the Vereinigte Leicht-metallwerke in Bonn or the Vereinigte Deutsche Metallwerke in Altena or Werdohl and of the Metallwerk Schwietzke, Düsseldorf (nonferrous metals)
- Trip round Ruhrort Harbour and visit to the model mine of the Gutehoffnungshutte

Saturday, June 11, Düsseldorf

Meeting of the Discussion Groups in the morning
- Materials Problems. Discussion Leader: Professor Dr. E. Houdremont
  - Manufacture of rails (cooling and heat-treatment, straightening, formation of corrugations)
  - Steel for deep drawing sheet (unkilled, semi-killed and killed steel, prevention of flow figures)
  - Organization of classification
- Thermal and Operational Economy
  - Importance of the technique of flow in the construction and operation of heating furnaces
  - Experiences with modern blast heaters
  - Advances in the technique of the cooling of heating and melting furnaces
- In the Nonferrous Metals Field (details to be arranged later)
  - Gases in metals
  - Induction heating for the heating up of ingots

Monday, June 13, Liege, Belgium

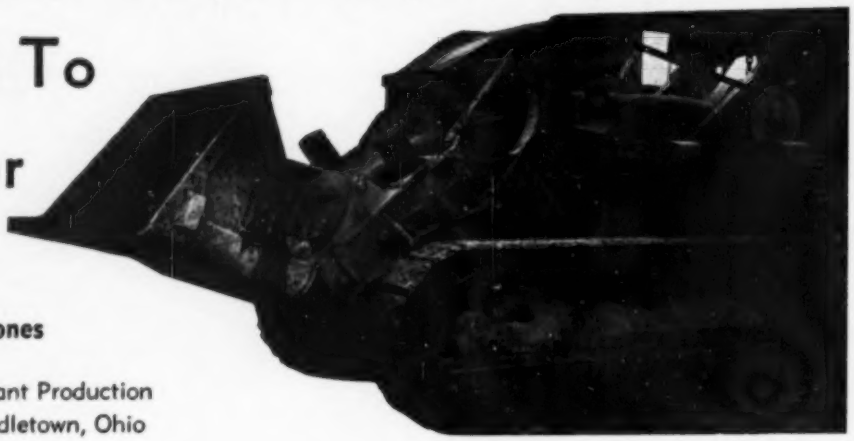
Travel from Düsseldorf to Paris
Optional stopover during day in Liege, Belgium
- Technical discussions and visits to works and laboratories, including the pilot plant of the low-shaft furnace, and laboratories, Centre National de Recherches Metallurgiques

June 14 to 19, France

Meetings of the Discussion Groups and technical visits in and near Paris to be arranged
- Lorraine, iron and steelworks and iron ore mining
- Lille District, iron and steelworks
- Central France, nonferrous metals, steelmaking
- Issoire and Savoy, aluminium and electric steelmaking
- Caen Steelworks

Final Plenary Session in Paris
Closing functions in Paris

Open Hearth Shops Adopt Developments In Aids To Labor

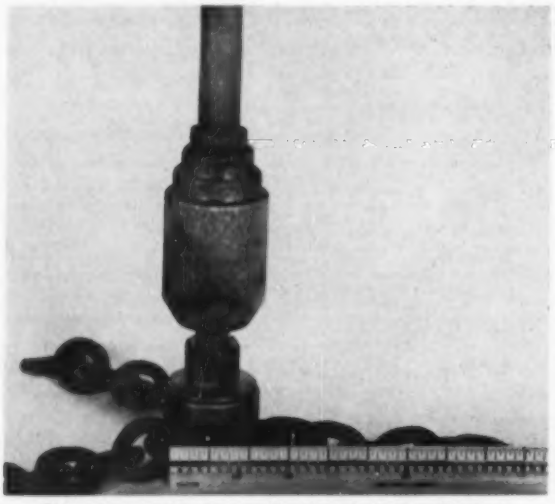


by

Vernon W. Jones

Superintendent, Steel Plant Production
Armco Steel Corp., Middletown, Ohio

THE open hearth shops in this country have, in recent years, made great advances toward aids to labor. The equipment used in this universal practice of lightening the load is familiar to most operators. However, there are some applications of new equipment that should be of interest to the industry as a whole. These pictures tell the story.



This apparatus does an excellent job of cleaning flue type checkers during furnace repairs. It is fastened to the rotating head of an air motor and dropped vertically into the checker openings.

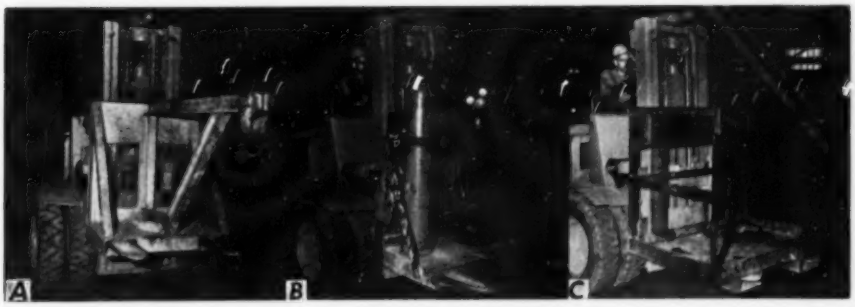
The rod lifter shown above does away with the old method in which the stopper rod maker lifted the assembly with his knee. It makes the entire operation less laborious and more efficient.

Belt conveyor loads material in overhead bins directly on the charging floor. This eliminates the problem of handling carloads which congest charging floor area.

Overshot loader is used for loading limestone and ore into buggies and removing slag from slag pockets. The machine loads slag directly into rubbish cars eliminating intermediate handling by cranes. This makes for better housekeeping in the repair area and contributes to safety.

Portable mixer is a help in speeding up the mixing of fireclay used in lining ladles.

Small buggy which runs on capping crane track is used to handle shot aluminum for mold additions.



Lift trucks are a universal tool. Three of the many uses are shown: A—Converted into light tractor crane, B—moving portable burning outfit, and C—transporting portable work platform.

General view of two 30-ton basic open hearths showing usual materials added in making a heat. Furnaces produce Grade B (0.28 pct C) steel used in making railroad castings.

Survey Provides Analysis of Cold Metal Shop Melting Practice

by S. L. Fredericks

DATA on the charging and fluxing practices in cold metal shops—more particularly those shops that use burnt lime and a low percentage of pig iron—were collected by submitting questionnaires to various basic open hearth shops.

The questionnaire submitted was divided into the following eight subjects: A—Size of heats tapped; B—average yield obtained; C—amount of pig iron charged in each heat; D—number of cuts of charge in each heat; E—amount of turnings used in each heat; F—types of substitutes for pig iron; G—type, size, and weight of burnt lime and method of application; and H—type and weight of fluorspar used.

Only those basic open hearth shops using low amounts of pig iron were requested to answer the questionnaire. Of the 32 shops contacted, the 19 or 60 pct that responded represented a fair cross-section of the U. S. To provide a good analysis and

S. L. FREDERICKS is Chief Chemist, American Steel Foundries, East St. Louis, Ill. This paper was presented at the AIME National Open Hearth Conference, Chicago, Apr. 5 to 7, 1954.

Table I. Charging Practice

Shops	No. Reported	Net Tons Tapped		Yield, Pct	
		Range	Avg	Range	Avg
Ingot	10	42 to 285	153.6	86 to 93	89.3
Casting	5	20 to 52	31.0	90 to 94	92.1

Only those basic open hearth shops using low amounts of pig iron were requested to answer the survey questionnaire. The average pig iron charged was 31.9 pct for ingot and 18.0 pct for casting shops.

comparison, the reporting companies were divided, according to their product, into ingot and casting shops. This breakdown shows that 14 ingot shops and five foundries make up the total of companies that returned the questionnaire.

Charging Practices

The information covering Items A and B in the questionnaire is shown in Table I. The average size heat reported was 123.6 tons in ingot shops and 31.0 tons in foundries. The ingot shop range was from 42 to 285 tons and the foundries from 20 to 52 tons.

The average yield on ingot shop heats was 89.3 pct with a range of 86 to 93 pct. Average casting shop yield was 92.1 pct with a range of 90 to 94 pct.

There are many factors affecting furnace loss. Various types of scrap promote various furnace losses due perhaps to its weather rusted condition as well as oxidation in the furnace during meltdown. Furnace condition such as excess air or an exposed loose charge may promote a loss. An average of 92.2 pct is usually used for calculating furnace weights and recoveries. This figure coincides with the average for foundries shown in Table I, but it is higher than figures for ingot shops.

Table II. Turnings Charged, Pct

Shops	No. Reported*	Turnings Charged, Pct	
		Range	Avg
Ingot	7	1 to 27	15.0
Casting	5	0 to 9	9

* Two ingot and four casting shops reporting use no turnings

It was surprising to find that many open hearth shops are using turnings or shavings rather extensively. In Table II, of seven ingot shops reporting, five report an average of 15.0 pct turnings in a charge, with a low of 1.0 and a high of 27.0 pct. Of five foundries reporting, only one uses turnings at a rate of 9.0 pct of total charge. The disadvantage of storing and using machine shop drillings, shavings, turnings, etc., is well known. In this day of a free and easy scrap market, other grades of scrap are readily available.

Table III. Pig Iron Additions

Shops	No. Reported	Iron in Total Heat, Pct		No. Cuts of Scrap and Iron	
		Range	Avg	Range	Avg
Ingot	10	17 to 45	31.9	2 to 7	4.1
Casting	5	11 to 25	18.0	1 to 2	2.0

Pig Iron Additions

The average amounts of pig iron used in foundries and ingot shops is shown in Table III. The average percent of pig iron used in 10 ingot shops reporting was 31.9 pct with a low of 17.0 and a high of 47.0 pct. The average foundry reporting used 18.0 pct pig iron with a range from 11.0 to 25.0 pct. Four hot metal shops turned in questionnaires, but this information was not used in making these comparisons.

An interesting observation may be made from this phase of the study. With pig iron selling for roughly $52.00 per net ton, the scrap market in the doldrums, No. 1 scrap selling below $30.00 per ton and in some localities as low as $21.00 per ton, a distinct saving can be made by charging a maximum amount of scrap and as little pig iron as possible. The use of substitutes for pig iron such as broken molds, graphite, and coke seems advantageous.

A variety of materials was used as a substitute for pig iron. In most cases some form of carbonaceous material was combined with additional scrap to make up for the reduction in pig iron. Tar, coke, graphite, cast iron, anthracite egg coal, broken

Table IV. Pig Iron Substitute

Shops	No. Reported*	Pig Iron Replaced, Pct		Cut in Which Substitute Is Charged	
		Range	Avg	Range	Avg
Ingot	10	0 to 100	51.8	1 to 3	2
Casting	5	17 to 100	41.1	1 to 3	2

* One ingot shop does not use pig iron substitute.

Table V. Lime Additions

Shops	No. Reported*	Lime per Ton of Charge, Lb Range	Avg
Ingot	10	110 to 150	120.9
Casting	5	100 to 129	113.8

* Combination of burnt lime and raw limestone is used by five ingot shops reporting.

molds, and coke breeze were listed as substitutes.

Besides the amount of pig iron used, it was of interest to find the manner of charging a heat; that is, the number of cuts necessary for completing the charge. The ingot shops required, on the average, four cuts to complete the charge with a minimum of two cuts and a maximum of seven cuts being reported. Four foundries reporting used two cuts to complete charging and one foundry used one cut. Of course, the ingot shops had the larger tonnage and required more cuts to finish the charge.

Table IV is a comparative study of the use of the above mentioned substitutes. This table shows the percentage of substitutes used and the position in the heat where they were used. The average amount of pig iron replaced in ingot shops was 51.8 pct of the total pig iron charge with one shop making no substitute. The highest substitution was 100.0 pct of the pig iron charge. The average substitution in foundries was 41.1 pct with a low of 17.0 and a high of 100.0 pct. One question that was not asked and which would be interesting to know is how often substitutes were used. The wording of the questionnaire was such that the reply may refer to only a possible emergency use.

Lime Addition

The questionnaire also requested information about the type of lime used, amount of lime charged, manner in which lime is used, and the chemical analysis specified for burnt lime.

Table V indicates that the average charge of lime in ingot shops is 120.9 lb per ton with a low of 110 and a high of 150 lb per ton of charge. Casting shops reported an average of 113.8 lb of burnt lime per ton with a low of 100 and a high of 129 lb per ton. Ten ingot shops and five foundries reported.

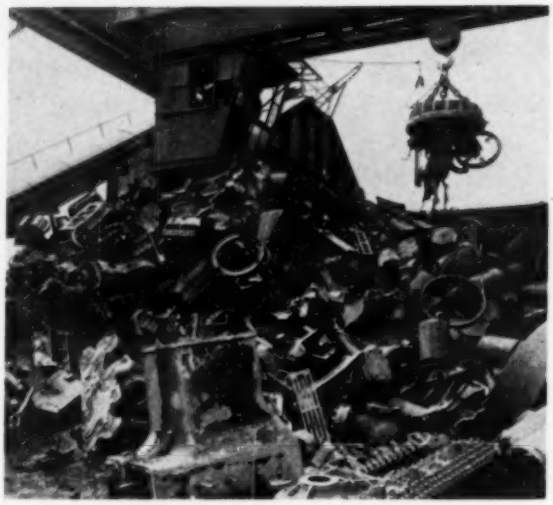
The five foundries used dead burnt lump lime exclusively. Five ingot shops used a combination of burnt lime and raw limestone. Three ingot shops used bagged pebble lime. One ingot shop reported using half burnt lime sized to approximately 6 in.

Table VI is a review of the chemical analyses specified for burnt lime. Two ingot shops and one casting shop have no chemical requirements for purchasing lime. The average chemical requirements for ingot shops are: 93.5 pct min CaO, 2.2 pct max $CaCO_3$, and 0.9 pct max SiO_2. The average requirements for foundries were 95.0 pct min CaO, 3.0 pct max $CaCO_3$, and 1.5 pct max SiO_2. All shops reporting chemical requirements were in close agreement.

Table VI. Chemical Analyses of Burnt Lime

Shops	No. Reported*	CaO, Pct Min	$CaCO_3$, Pct Min	SiO_2, Pct Max
Ingot	10	93.5	2.2	0.9
Casting	5	95.0	3.0	1.5

* One casting and two ingot shops have no burnt lime specifications.



A distinct saving may be made in cold metal shops by charging a maximum amount of scrap. Pig iron sells for approximately $52 per ton and scrap between $21 to $30 per ton.

Each open hearth was also asked to describe the manner in which lime is used. All 15 shops reporting used the same procedure for initial lime addition during the charging operation. The lime, either burnt or raw limestone, was added after a cut of light scrap had been used to cover the bottom. Most shops did not make further additions except in emergencies. Once lime-silica rations had been established for a given percent reduction of sulphur and phosphorus those weights were maintained.

Table VII. Fluorspar Requirements

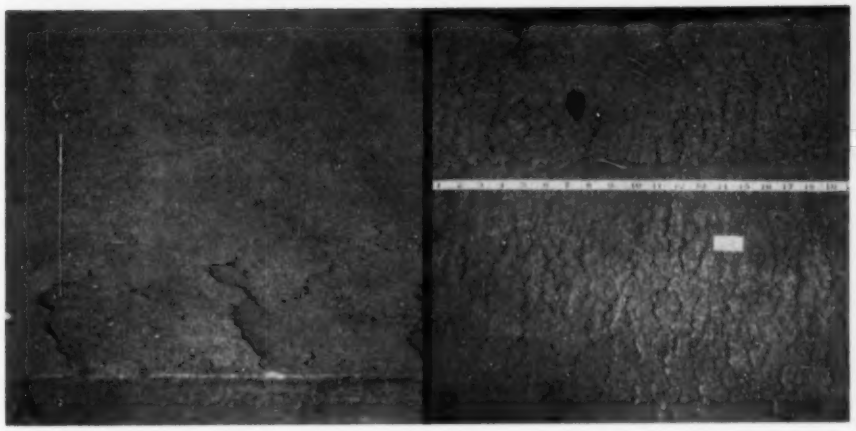
Shops	No. Reported	Fluorspar, Lb Per Ton of Charge Range	Avg	CaF_2, Pct Range	Avg
Ingot	10	1 to 15	7.2	70 to 80	76.6
Casting	5	4 to 14	9.0	70.0	70.0

Of 15 shops reporting, five shops used feed lime for shaping up every heat, three shops used feed lime when preliminary analysis indicated additional lime was necessary for sulphur control, and one shop reported using the slag potty test for determining the condition of the slag.

Fluorspar Additions

To round out the survey of charging and fluxing practices a request was also made for information concerning the use of fluorspar. Of 10 ingot shops reporting, nine shops purchased fluorspar according to specifications. The average percent of minimum calcium fluoride specified was 76.6 pct with a range of 70.0 to 80.0 pct as shown in Table VII. The foundries each required a minimum of 70.0 pct calcium fluoride in fluorspar.

The amount of spar used varies somewhat. The ingot shops averaged 7.2 lb of spar per ton of charge with a range of 1.0 to 15.0 lb per ton. The casting shops averaged 9.0 per ton of charge with a range of 4.0 to 14.0 lb per ton.



Hot rolling defects in stainless steels are shown as follows: A—Defect characteristic of unbalanced 18-8 Cr-Ni austenitic steels, and B—hot shortness of higher alloyed Cr-Ni austenitic stainless steel.

Rare Earths Counteract Hot Rolling Defects in Stainless Steel

by R. H. Henke and R. A. Lula

CONVERSION of a stainless steel ingot into a hot rolled semifinished product has always been a source of some difficulty. During the early days of the stainless steel industry most of the products were from a hand sheet mill or plate mill where small ingots could be used. However, as the industry grew so did the ingot size, and with this growth problems arose concerning ingot conversion.

The rollability of stainless steel is determined by metallurgical factors and the type of equipment available. The metallurgical factors which will be discussed here include the structure and grain size of the ingot (which are functions of the ingot size), pouring temperature, influence of various small additions, and whether the alloy has solidified in the ferritic or austenitic phase.

Ferritic Stainless Steels

Types 430 and 446 stainless are substantially ferritic but small amounts of austenite can form at the rolling temperature. The dynamic resistance to deformation of the three types of stainless steel and of plain carbon steel at 2150°F is shown in Table I.

Since the testing speed in dynamic testing is of the same order of magnitude as the rate of deformation in hot rolling, the flow stresses listed in Table I are indicative of the resistance to deformation during rolling for the various steels. The resistance to deformation of the ferritic steels is low and they roll easily without the use of special addition agents such as rare earth metals or oxides. When difficulties are encountered in rolling the 27 pct Cr steel they, in general, can be related to coarse ingot grain structures or to the formation of small amounts of austenite at the rolling temperature.

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It is generally agreed that duplex structures in stainless steels promote susceptibility to hot shortness at rolling temperatures and is to be avoided. The effect of austenite formation on high temperature ductility is readily shown by hot twist tests which provide a convenient method of evaluating the hot working properties of stainless steels.

The specimens employed in the test were ⅝-in. diam bars 30 in. long with 11 in. in the hot zone. The speed of rotation was 200 rpm. The ductility as measured by the hot twist test shows a sharp decrease coincident with the formation of austenite. Elimination of nitrogen and alloying with a sufficient amount of a ferritizing element, i.e. titanium or columbium precludes formation of austenite and the decrease in the ductility curve is eliminated. The influence of the amount of austenite on ductility is shown by the curves in Fig. 1. The influence of titanium goes beyond the elimination of austenite since the increase in ductility goes far above the other ferritizing elements such as Cb, Si, V, etc. Titanium and columbium also seem to have a grain size refining influence on the structure of the ingots.

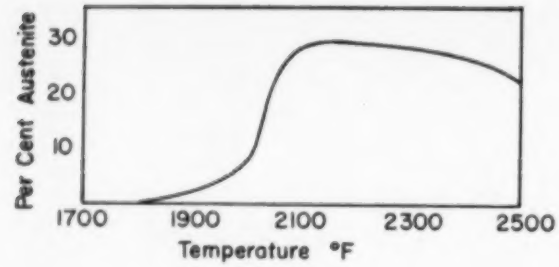
Martensitic Steels

Depending upon the balance of the specific chemical elements, the 12 pct Cr steels can be either austenitic or ferritic or have a duplex structure at the

Table I. Dynamic Resistance to Deformation of Various Steels

Type of Steel	Resistance Psi
Carbon steel* (0.05 pct C)	14,100
410	23,900
430	18,250
304	35,900

* Werkstoffhandbuch Stahl und Eisen EL-1.



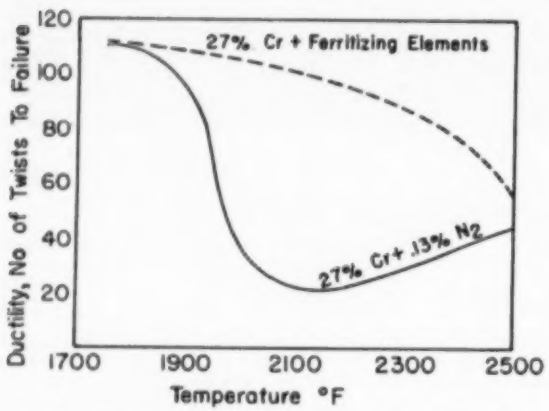


Fig. 1—Relationship is shown between austenite formation and hot ductility for Type 446 (27 pct Cr) steel.

rolling temperature. Their hot rolling behavior is dependent upon the type of structure. If they are austenitic the resistance to deformation is somewhere between the ferritic and the austenitic grades in Table I. Type 405 stainless is made ferritic by aluminum addition and rolls well. Types 403 and 410 with carbon in the upper range and chromium in the lower range of analysis are austenitic and also roll well. The 12 pct Cr steel with carbon in the lower range and chromium in the upper range has a duplex structure and its characteristic hot shortness.

Austenitic Steels

The austenitic chromium-nickel stainless steels are quite different in their behavior from the plain chromium steels. For one thing, they have considerably higher resistance to deformation which is in agreement with the higher creep and stress rupture properties of these steels.

Hot rolling operations with low speeds of deformation allow long periods for recrystallization and are therefore easy to accomplish. One of the hot working difficulties of austenitic stainless steels, especially those of high alloy content, may be related to the slower rate of recrystallization in comparison with lower alloyed steels. This becomes more evident at the high rate of deformation of the modern rolling mills.

Table II. Analyses for Alloys Shown in Isothermal Section, Fig. 3

Designation in Phase Diagram	Percent Cr	Percent Ni	AISI No.
A	16 to 18	6 to 8	301
B	17 to 19	8 to 10	302, 303
C	18 to 20	8 to 11	304
D	22 to 24	12 to 15	309
E	24 to 26	19 to 22	310

Table III. Composition of Type 304 Stainless Steel

Analysis	Pct
C	0.065
Mn	1.55
P	0.035
S	0.023
Si	0.57
Cr	18.60
Ni	9.42

In Fig. 2 the hot working properties of some of the most important types of stainless steels are given as measured by the hot twist test. These results represent average values of a very large number of tests. Type 321 has by far the highest ductility which, no doubt, is because of the titanium addition and is followed by the plain 18 Cr-8 Ni steels (Types 301, 302, and 304). Types 310, 316, 317, 309 and austenitic Cr-Mn steel (15 Cr, 15 Mn, 1 Ni) have somewhat lower ductilities.

Two additional factors may detract the workability of austenitic stainless: hot shortness due to a duplex structure and an inherent hot shortness characteristic of single phase high alloyed Cr-Ni steels. Duplex structures can occur in most of the grades. The hot shortness due to duplex structure can be avoided by proper balance of elements, selection of a suitable hot rolling temperature and a good soaking pit practice. The grades subject to this type of hot shortness are 301, 304, 316, 321, 309, and austenitic Cr-Mn steels. The type of hot shortness occurring in the higher alloyed steels is characteristic for Types 310, 309, 316, 317 and austenitic Cr-Mn steels. A noticeable improvement in this second type condition can be obtained by the use of rare earths elements.

Duplex Structure

The ternary phase diagrams in Figs. 3 and 4 are adapted from the work of Pugh and Nisbet[1] for Fe-Ni-Cr alloys with very little impurities. The cross hatched curves represent the phase boundaries for commercial alloys: c-d is the alpha + gamma/

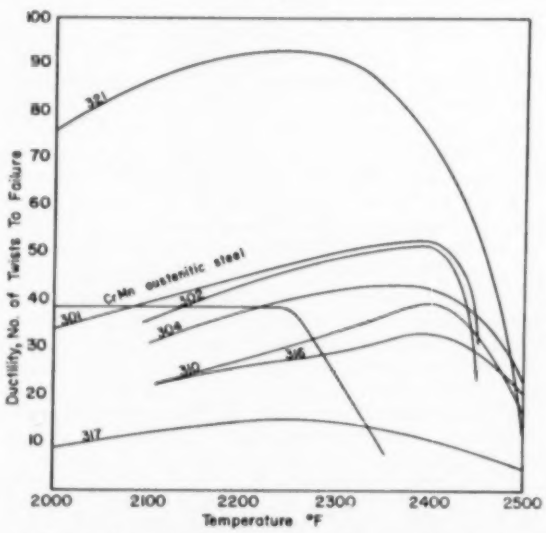


Fig. 2—Average values of hot ductility vs testing temperature of austenitic stainless steels are given as measured by hot twist test.

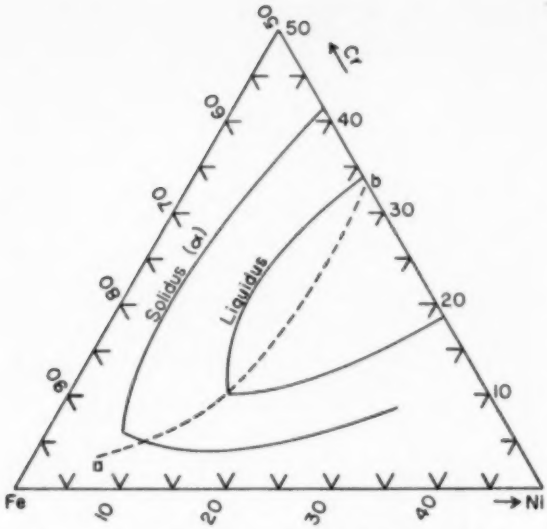


Fig. 3—Ternary phase diagram for Fe-Ni-Cr alloys adapted from Pugh and Nisbet[1] indicates solidus and liquidus at 2642°F.

gamma phase boundary at 2012°F and e-f the same boundary at 2372°F.

As seen in Fig. 3, upon solidification the first solid particles will be ferrite for compositions to the left of the line a-b (the projection of the intersection of the two liquidus surfaces) and austenite for compositions to the right of a-b. The composition of the solid alpha will be higher in chromium and lower in nickel than the composition of the alloy, resulting in segregations which will extend the presence of ferrite in as cast material beyond the temperature and average composition of the alloy.

The isothermal section in Fig. 4 shows that at 2012°F only the lower alloyed 301 can be partially ferritic while all the others are completely austenitic.

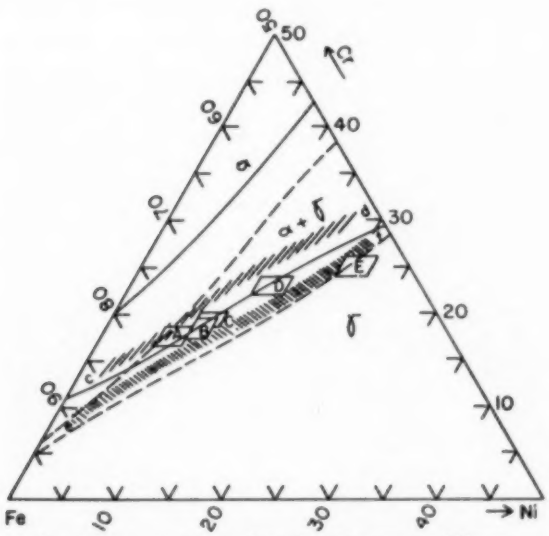


Fig. 4—Lines used in isothermal section are as follows: ——— at 2012°F, — — — — at 2372°F, ////// at 2012°F for commercial alloys, and \\\\\\ at 2372°F for commercial alloys. Table II gives the analyses for alloys A, B, C, D, and E.

At higher temperatures, however, the alpha and gamma field is displaced to the right and some of the 18-8 Cr-Ni steels are in the alpha + gamma field at 2380°F. Due to segregation, however, ferrite can be found in all the alloys located on the left of the curve a-b when they are in the as cast condition. The ferrite resulting from segregation can be eliminated or reduced to its equilibrium amount by proper soaking pit practice.

Fig. 5 shows the effect of time in decreasing the amount of ferrite in a Type 304 stainless steel at 2380°F, the soaking pit temperature of this type. The composition of this heat is given in Table III.

Small specimens from this heat were taken from the center of a 17x53x75-in. ingot, weighing 15,000 lb. After heat treatment the amount of ferrite was determined by the Magna-gage. At 2380°F the amount of ferrite decreased from 12 pct in ½ hr to 4 pct in 6 hr and 3.3 pct in 14 hr. At 2300°F it dropped from 9.5 pct in ½ hr to 1.6 pct in 14 hr.

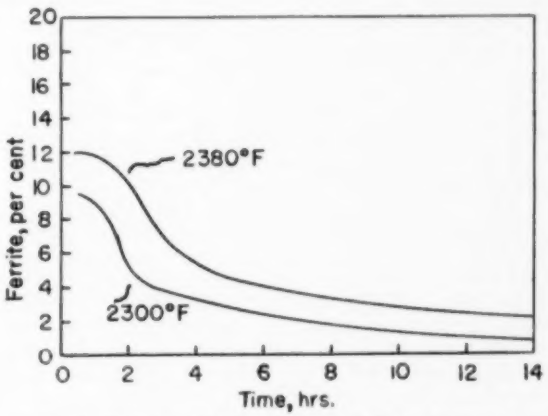


Fig. 5—Variation with time of amount of ferrite in as-cast 18-8 Cr-Ni steel is shown.

Hot working eventually completes the job of eliminating segregation and the final products are completely free from ferrite when properly annealed. Fig. 6 shows the as-cast structure in the center of the ingot. Fig. 7 shows the structure after ½ hr at 2380°F with about 12 pct ferrite forming an almost continuous network. Only 5 pct remains after 6 hr at 2380°F as shown in Fig. 8.

The curves in Fig. 5 show that the amount of ferrite can best be reduced by soaking at temperatures somewhat below the actual rolling temperature of 2380°F. For this reason, unbalanced 18-8 Cr-Ni heats that are difficult to roll because of excessive amounts of ferrite are subjected to a special heating cycle; whereas balanced steels are heated directly to 2380°F, soaked for a certain period of time as determined by the ingot size and then rolled.

The following formula is used to determine the balance of 18-8 Cr-Ni heats:

$$30C + \frac{Mn}{2} + Ni + 11.8 - 1.3\,Cr = A$$

If A, the balance factor, is less than −1, the heat is considered unbalanced and the ingots are soaked at 2200°F in order to reduce the segregation and the amount of ferrite as much as possible before heating for a short period at 2380°F. If A is greater than −1, the ingots are heated directly to 2380°F.

Fig. 6—As-cast structure is shown from the center of Type 304 stainless steel ingot, etched with NaCN then oxalic acid (electrolytic) and magnified 100X.

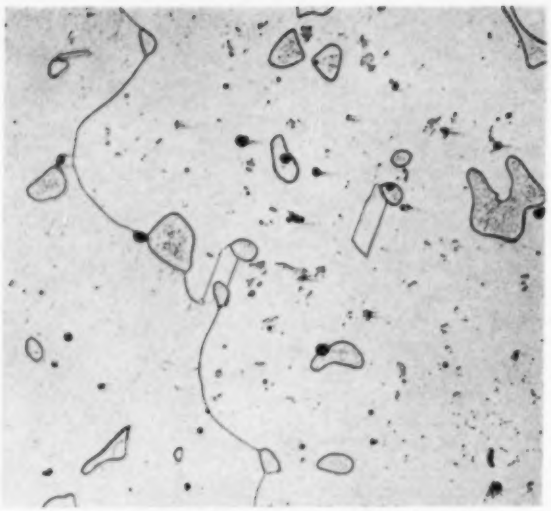


Fig. 8—Structure of Type 304 stainless steel after 6 hr at 2380°F followed by water quench, etched with NaCN then oxalic acid (electrolytic) and magnified 100X.

Experiments at Allegheny Ludlum show that a small percentage of ferrite is not necessarily detrimental to hot rolling but large percentages, especially when distributed in a network, can cause serious hot shortness in rolling. The mechanism of failure seems to be related to the rather large difference in strength and ductility of austenite in comparison to ferrite. In the initial stage of rolling when the temperature is around 2300° to 2400°F both ferrite and austenite have similar low flow stresses, but as the temperature drops only the austenite strengthens while the ferrite remains soft causing rupture due to excessive deformation in the ferrite. Fig. 9 shows the rupture produced by a ferrite network in a slab of 18-8. The sharp drop in ductility in Fig. 2 for several grades of steel is related to the formation of ferrite.

Inherent Hot Shortness

The higher alloyed stainless steels, Types 309, 310, 316, 317, and austenitic Cr-Mn steel are characterized by an inherent hot shortness at the hot rolling temperatures. The severity of the hot shortness increases with the size of the ingot. For this reason it is believed that hot shortness, if not caused by grain coarseness, is at least related to it.

The first method of overcoming this type of hot shortness is by the addition of *grainal*, a nucleating deoxidizing ferroalloy whose analysis is given in Table IV. A total of 8 lb per ton, four in the furnace and four in the ladle, improves the hot workability of the higher alloyed stainless steels. This is particularly true if the ingot size is less than 8000 lb. The inoculating and the deoxidizing action of the *grainal* may account for the improvement in hot working.

In recent years the rare earths have been used successfully to improve the workability of austenitic steels at high temperatures. The rare earths are marketed in the metallic (mischmetal) or oxide form. Both can be used.

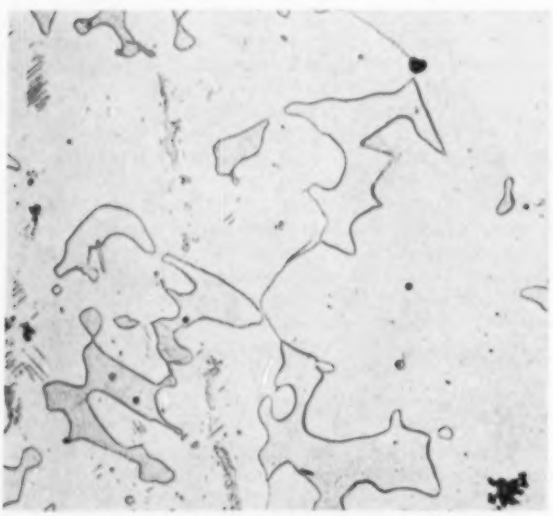


Fig. 7—Structure of Type 304 stainless steel is shown after ½ hr at 2380°F followed by water quench, etched with NaCN then oxalic acid (electrolytic) and magnified 100X.

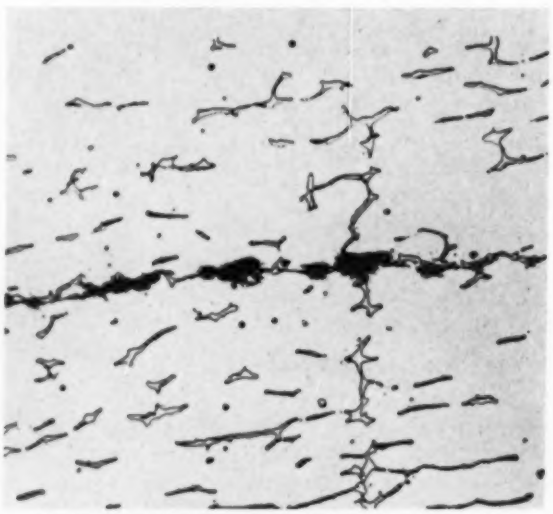


Fig. 9—Internal rupture due to ferritic network in a slab of 18-8 Cr-Ni steel etched with NaCN is shown. Reduction in ductility is related to ferrite formation.

Metallic rare earths in amounts from 2 to 5 lb per ton of steel were added in the ladle in initial experiments with the rare earths. It was found that at least 3 lb per ton were necessary for improvement in workability, and maximum improvement could be attained by the addition of 4 to 5 lb per ton. Ingots of Types 309, 310, 316, 317, and austenitic Cr-Mn steels that had been treated with the proper amount of rare earths could be easily rolled in one single conversion, whereas a double conversion (consisting of slabbing, conditioning, and rerolling) was sometimes required prior to the use of rare earths. The workability correlates with the amount of rare earths added rather than the residual amount retained in the steel.

The metallic rare earths, however, impair the cleanliness of the steel with the impurities being localized close to the outside surface of the ingot.

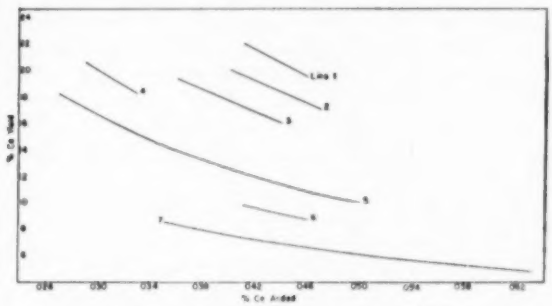


Fig. 10—Percent cerium yield as a function of cerium added in Type 316 stainless steel is shown. Lines 1 to 7 correspond to various charges and melting practices.

These impurities give rise to a surface defect called *hairs* in finished sheet material and cause cracking during upsetting of bars. These facts, as well as economic considerations, led to experiments with additions of rare earth oxides which proved successful in improving the workability and the cleanliness of the steel.

The oxides of the rare earth metals are added in the ladle. An amount of 3 lb per ton of steel is added if the melt is deoxidized with *grainal* or 5 lb per ton when no *grainal* is used. Fig. 10 shows the cerium yield for a large number of Type 316 heats made in 10 and 25-ton furnaces.

Cerium is the easiest to analyze and constitutes the largest percentage in the rare earth oxides making it the most convenient to use. It can be seen first that the larger amount added the smaller the percent cerium yield.

Rare Earths and Rollability

In trying to correlate the rollability with the amount of rare earths, the percentage yield from an ingot was measured at a certain stage of production. For Type 316, Fig. 11 shows that there is an optimum amount of about 0.036 to 0.040 pct added cerium corresponding to a rough range of 2.5 to 3.0 lb per ton of rare earth oxide addition. In Type 316 this relationship has a simple correlation coefficient significant at the 1 pct level of statistical assurance. Type 310 shows the same general tendency for cerium yield and ingot yield but the correlation is not as good as in 316 steel. There also seems to be a general tendency for the heats with higher retained cerium in the ladle to have higher yields.

Table IV. Composition of a Nucleating Deoxidizing Ferroalloy

Analysis	Pct
Ti	15 to 20
Al	10 to 13
V	0 to 25
Si	3 to 5
Mn	0 to 8
B	0.2 to 0.5
Zr	0 to 4
Fe	balance

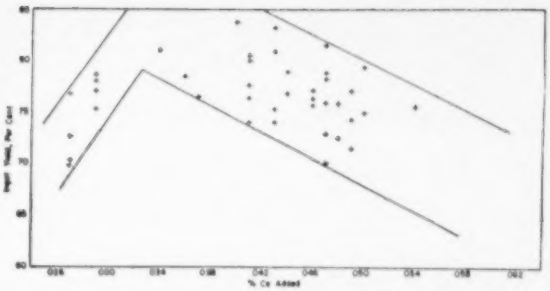


Fig. 11—Percent ingot yield as a function of percent cerium added in Type 316 stainless steel indicates an optimum amount of about 0.036 to 0.040 pct added cerium.

The percentage ingot yield was improved by the use of rare earth oxides. Fig. 12 shows the frequency of ingot yield of Type 316 prior to the adoption of rare earths in 1951 and in 1953 when rare earths oxide was used. The average was 70.4 pct for 1951 and 73.8 pct for 1953. The favorable influence of rare earths is even more evident if instead of using all the heats selected only those with 0.035 to 0.045 pct cerium addition are considered. In this case the average ingot yield is 78.3 pct. The frequency plot is seen in Fig. 13. Besides the increase in yield there are further advantages because the heats treated with rare earths can be rolled in one single conversion as compared to the more expensive double conversion.

The improved rollability of the austenitic steels treated with rare earths appears to be caused by increased ductility and lower flow stresses at the roll-

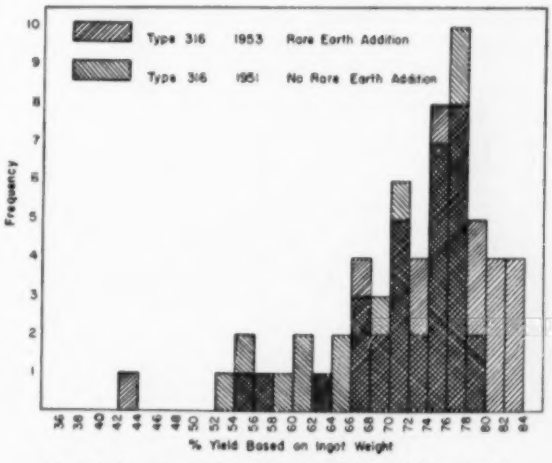


Fig. 12—Frequency of yield for Type 316 stainless steel is shown for 1951 prior to the adoption of rare earths and for 1953 when rare earth oxides were used.

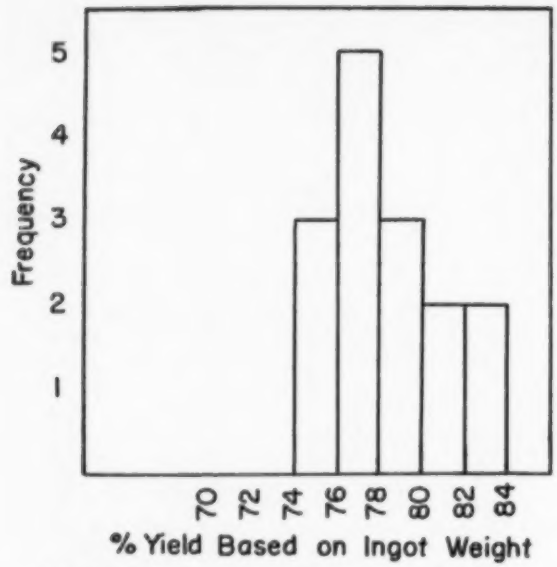


Fig. 13—Frequency of yield for Type 316 heats with 0.035 to 0.045 pct cerium added as rare earth oxides gives an average ingot yield of 78.3 pct.

ing temperature. Fig. 14 shows the ductility of a production heat of Type 310 before and after the rare oxides were added. The material for specimens was taken as 60-lb ingots. Fig. 15 shows the average ductility of several heats of austenitic Cr-Mn steels with and without rare earths addition. In both cases it can be seen that the ductility is considerably higher for the treated material. The Cr-Mn steels are the only grade which show consistently higher ductility after rare earths addition while occasional reversed results are obtained with other grades.

Numerous experiments have been performed on laboratory and production heats in an effort to uncover the mechanism by which the rare earths improve the hot working properties, and also to determine what effect rare earths might have on other properties. The results of these studies have been inconclusive and no mechanism to account for the effect of the rare earths can be offered.

Part of the difficulty arises from the fact that although positive improvement in the hot working of large production heats is obtained, there are a large

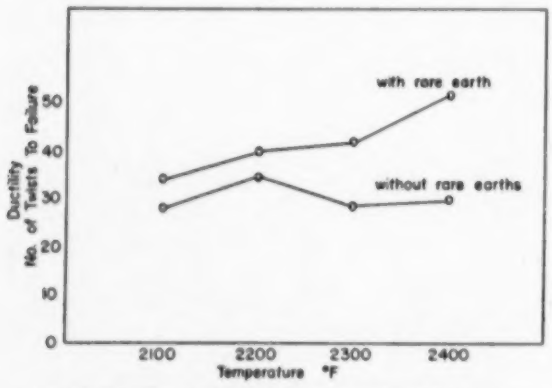


Fig. 14—Ductility of Type 310 stainless steel heat increases after rare earth additions.

number of variables involved and the results are mostly obtained statistically. Further complications result from the fact that very little, if any, influence of rare earth addition is ever found on the properties of small laboratory heats.

Summary

The behavior of the three groups of stainless steels (martensitic, ferritic, and austenitic) with respect to the conversion of ingots into hot rolled semifinished products can be summarized as follows:

A—The martensitic and ferritic steels roll easily and do not require rare earths additions.

B—Slight difficulties are encountered in the 27 pct Cr steel because of austenite formation. This can be overcome by the addition of ferritizing elements.

C—Austenitic steels are more difficult to hot roll and are characterized by two types of hot shortness; one due to a duplex ferrite and austenite structure, and one inherently associated with the higher alloyed austenitic steels.

D—Hot shortness because of a duplex structure can be eliminated by proper soaking pit practice and adequate balance of composition.

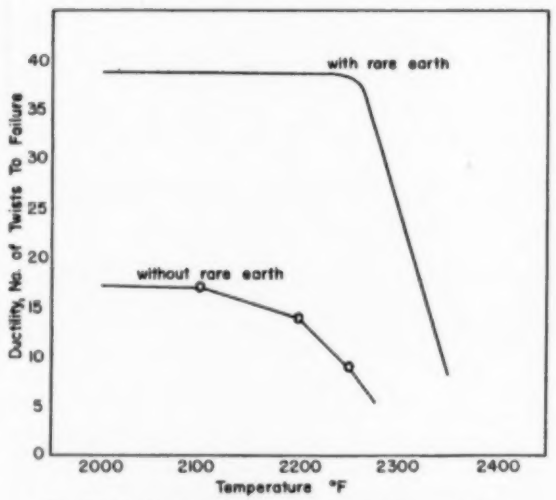


Fig. 15—Cr-Mn austenitic stainless steel increases in ductility after rare earth additions.

E—The inherent hot shortness of higher alloyed steels can at present be best counteracted by the addition of rare earths.

F—There seems to be an optimum amount of rare earths oxide addition expressed as 0.036 to 0.040 pct cerium.

G—Both metallic rare earths and their oxides can be used successfully, but the use of oxides has become the present practice at Allegheny Ludlum. Ingots that had to be rolled in a double conversion can now be rolled in one single conversion and the actual ingot yield has been raised.

H—Improvements produced by rare earths addition appear to be related to increased ductility at the rolling temperature. The exact mechanism is not very well understood.

Reference

[1] J. W. Pugh and J. D. Nisbet: Iron-Chromium-Nickel Ternary System. *Trans.* AIME (1950) **188**, p. 268.

International Nuclear Engineering Congress Highlights Metallurgical Research

by Joseph H. Bach

THE widespread effects of nuclear engineering on our way of life have never been more clearly shown than at the First International Nuclear Engineering Congress held under the auspices of the AIChE at the University of Michigan, Ann Arbor. The technical community has of course been aware of the impact of this new field for some time, but well attended sessions on such nontechnical subjects as nuclear engineering and law indicated how much civilization has become involved with the *atom*.

There was probably no more logical place to hold such a meeting than the campus of the University of Michigan whose Phoenix Project has been set up to study the effects of nucleonics on every field of human endeavor. Under the slogan *Atoms for Peace* an exposition was held in conjunction with the congress; 20 commercial exhibitors were represented and at least four offered to build complete research reactors for anybody who could obtain AEC authorization.

Atomic Energy Abroad

The congress was truly international with more than 1100 delegates from 22 nations and the U.S. attending. Many were high level scientists and administrators of installations in the various countries equivalent to our AEC.

Relatively few nations outside of the U. S. and Russia have reactors today. England has a number of research reactors and presumably some weapons production units and is at present primarily interested in power reactors to solve the island's power shortage. Canada has two reactors and is building a third one; and France has two research reactors which are also used for power development study. Sweden is running a heavy water pile which has the interesting facet of being completely underground. The Netherlands and Norway are operating a joint reactor near Oslo, with one immediate aim the development of naval reactors for the Norwegian Merchant Marine. Belgium, India, and Spain do not have any units yet but are definitely planning on building some within the near future. India is potentially one of the largest users of atomic power and hopes to exploit its rich thorium sands for this purpose.

The importance of metallurgy to the whole field of nuclear engineering was shown by the fact that out of 90 technical papers presented at the Congress 15 concerned themselves with metallurgical problems, and 10 more dealt with such subjects as liquid metals, corrosion, and radiation damage.

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Raw Materials

The economic exploitation of natural resources is of prime importance to any nation interested in nuclear energy and many papers dealt with this subject. *The Extractive Metallurgy of Uranium* was discussed by R. D. Macdonald of Battelle Memorial Institute who showed that leaching of the deposits with acids or caustic has resulted in the best yields since the uranium-bearing ores are widely dispersed in microscopic particles. Three types of ores are now being exploited: primary ore such as pitchblende; oxidized deposits such as carnotite found on the Colorado plateau; and refractory ores. The latter were of no importance until recently when large amounts of davidite were found in Australia. It seems that the best method for beneficiation lies in the fact that the uranous (U^{+4}) ion is almost insoluble as opposed to the high solubility of uranyl (U^{+6}) ion.

J. VanImpe of the Nuclear Energy Research Study Center in Brussels, Belgium, discussed the *Preparation and Fabrication of Pure Metallic Uranium* and evoked general surprise when he stated that the country that owns the world's richest uranium deposits still refines this metal in 40 lb pilot plant batches. The hydrate is converted to UF_4 by successive calcination, fluorination, and vacuum decomposition. The tetrafluoride is then reduced in a vessel lined with CaF_2 and the metal is further refined by vacuum casting. The resultant uranium is of excellent quality having a density of 19.02 g per cu cm and totaling less than 175 ppm in impurities with a yield of 98.5 pct or better.

The economic value of uranium was pointed up when G. E. Brown of the Mallinckrodt Chemical Works discussed *A Process for Recovery of Uranium from Pitchblende Digestion Residues*. These residues, which are left after treatment of pitchblende with nitric acid, are called gangue lead cake and contain from 0.5 to 5 pct U in the form $UO_2 \cdot Mo_2O_7$ from which uranium is extracted by carbonate leaching.

The importance of thorium for breeder reactors is now definitely recognized and two different processes are under investigation for the most efficient extraction of this element from monazite sands. M. Smutz of Iowa State College spoke on *The Ames Process for Separation of Monazite* in which the sands are leached in sulphuric acid at 200°C; successive stepwise raising of the pH removes the UO_2 and rare earth impurities. Another process was reported by R. B. Filbert of the Battelle Memorial Institute who described a *Process for Recovering Thorium and Rare Earths from Monazite* by digestion of the sands in hot caustic followed by acid precipitation of the rare earths. The succeeding discussion brought out

the fact that full scale plants using the latter process are in operation both in Brazil and in India.

Reactor Fuels and Construction Materials

The metallurgical problems associated with reactor construction have presented a real challenge to the physical metallurgists, primarily because most of the materials having acceptable nuclear characteristics were considered laboratory curiosities until a few years ago; thus little background information is available for such metals as Be, Zr, Th, and U.

Preparation, Properties and Uses of Beryllium was discussed by E. J. Boyle of the Oak Ridge National Laboratories who showed that in spite of the large potential demand for this metal as a moderator, metallurgical difficulties and high costs have kept it from being used extensively. Vacuum castings produce billets with columnar structures which must be broken up by severe working. This is usually done by forging or extracting in a mild steel sheath above 1700°F. Best results are obtained by hot pressing beryllium powder above 2000°F followed by hot extrusion. The resultant metal is however anisotropic with its best mechanical properties in the working direction.

The conventional aspects of the powder metallurgy of beryllium were treated by H. H. Hausner and H. B. Michaelson of Sylvania Electric Products, Inc., in a paper entitled *Powder Metallurgy of Zirconium and Beryllium*. They showed that isotropic ductility is obtained by cold pressing and vacuum sintering. This metal can be cold worked up to 10 pct and a 1000°C anneal will remove all traces of such work permitting further reduction. The process is, however, noneconomic since vacuum sintering causes evaporation of up to 50 pct of the metal; sintering in argon prevents this but also results in lower density material.

The same paper discussed the powder metallurgy of the newest structural metal, zirconium. Zirconium powder can be cold pressed and vacuum sintered but the ductility of the wrought metal made it difficult to comminute so that hydriding was tried. Zirconium is converted to ZrH at 800°C and then to ZrH_2 at 400°C; this compound is brittle and easy to break up and is reduced back to Zr in a vacuum at 700°. Experiments have shown that better final densities are obtained if the hydride is reduced and sintered in a single operation and grain growth-density correlations showed that low density compacts tend to have fine grained structures, the holes suppressing the grain growth.

Melting and Fabricating of Zirconium was discussed by J. W. Holladay of Battelle Memorial Institute. The reduction of $ZrCl_4$ by the Kroll process now produces all the Zr for reactor uses and the iodide process has been abandoned as uneconomical. The sponge which results from the Mg reduction is arc melted in an inert atmosphere using a consumable electrode. The ingots can be hot worked in air but must be kept below 1550°F since the high temperature (BCC) phase has a high gas diffusion rate and the metal is sensitive to nitrogen and hydrogen impurities.

For extrusion Zr must be sheathed but otherwise it can be handled without trouble and its machining and joining properties are quite similar to those of aluminum. Discussion brought out that the hafnium separation is only necessary for nuclear applications of the metal and the impurity does affect any of its other properties appreciably.

Corrosion of Zirconium in High Temperature Water was discussed in the first declassified paper ever issued on this subject by D. E. Thomas of the Westinghouse Electric Corp. The metal is extremely sensitive to surface preparation and corrosion proceeds by a slow buildup of ZrO_2 which suddenly becomes nonadherent, at which point corrosion proceeds exponentially. Most of the tests were run in 600°F water using zirconium made by the iodide process. Peculiarly, the water purity does not seem to be an important factor, but very small amounts of nitrogen and carbon in the metal increase the corrosion rates considerably. It was mentioned that due to improvements the Kroll process now produces zirconium whose corrosion resistance approximates that of the iodide material.

The metallurgy of the actual reactor fuels was reviewed in two papers on uranium and thorium, respectively. With respect to the first, F. A. Rough of Battelle Memorial Institute discussed the *Metallurgy of Uranium* and stated that in spite of its high oxidation potential the metal can be worked by any of the common methods, but that cold reduction is limited and frequent annealing necessary. Considerable progress has been made in determining phase diagrams of binary alloys. Although uranium has a marked tendency to form intermetallic compounds, such metals as Zr, Ti, Mo, and Cb show extensive solid solubility at the uranium-rich end. There is also some indication that the first three of these improve both the mechanical and corrosion properties of uranium.

Thorium Metallurgy was discussed by J. R. Keeler, also from Battelle Memorial Institute. The metal is refined by a reduction process quite similar to that of uranium and yields clean massive ingots. The metal has an FCC low temperature phase, and evidence of a BCC phase above 1440°C has been found. Although it must be protected from the atmosphere during heating, it is easy to work, and so ductile that it has been reduced 95 pct with the intermediate annealing. Thorium is extremely sensitive to carbon and 0.25 pct C increases its tensile strength from 20,000 to 60,000 psi reducing its elongation from 60 to 25 pct. A bad feature is its low corrosion resistance to hot water, but this can be remedied since thorium is easily electroplated.

The effects of radiation damage on structural materials constitute a major problem in reactor design, but this seems to be one field in which the metallurgists need not fear the competition of the plastics industry. V. P. Calkins of the General Electric Co. spoke on *Radiation Damage to Nonmetallic Materials*, and, comparing these with metals, came up with a factor of 1000 in favor of the latter.

Radiation Effects on Structural Materials was discussed by C. R. Sutton of the Argonne National Laboratory. He mentioned that such damage is characterized by three points: A—It is localized and microscopic; B—It is nondirectional; and C—It is much less severe than cold work. Fatigue, impact, and tensile tests on steels and high temperature alloys have shown that the effect of nuclear bombardment are characterized most often by increases in hardness and tensile strength with accompanying loss in ductility. In no case, however, was the damage so severe that proper design could not compensate for it.

Ingot Cracks in Killed, Fine-Grained C1020 Steel

by M. A. Orehoski, N. R. Arant, and J. A. Pusateri

Plant studies on commercial-size ingots and laboratory experiments with induction furnace heats have demonstrated that the major source of ingot cracks is associated with two conditions arising during top-pouring practice: 1—solidification during pouring, and 2—turbulence created by the impact of the stream. Methods of controlling the two factors were effective in eliminating or significantly reducing ingot cracks.

BECAUSE the process of removing surface defects from hot-rolled product is so costly, the steel industry is striving constantly to develop methods of preventing, or at least decreasing, the occurrence of surface defects. Investigations have revealed steelmaking and processing variables related to major surface defects, and controlling these variables has led to improvements in surface quality. However, the fundamental causes of major surface defects, such as ingot cracks, have not been determined; consequently such defects persist.

At the Research and Development Laboratory of the United States Steel Corp. in Pittsburgh, a seam research program was initiated to determine the fundamental causes of certain major defects. As a part of the program, ingot cracks in killed, fine-grained C1020 steel were selected for study. A cost survey indicated that, of the steels produced in sizable tonnages, carbon steels in the range of 0.18 to 0.23 pct C content require the most conditioning. Since this is particularly true of C1020, any improvement of surface quality that might be effected from the study would be beneficial. Also, since the frequency of ingot cracks is exceedingly high for this grade, the steel would provide an excellent opportunity for a thorough study of the ingot-cracking problem. Why steels in this carbon range tend to exhibit more ingot cracks than do other steels was not considered in the investigation; the mechanism of ingot-crack formation was of paramount importance. Also, only the top-pouring practice was considered in the investigation, because this pouring procedure is used more extensively than others.

In this study, ingot cracks are defined as deep surface defects that sometimes are observed in an ingot prior to rolling but usually are observed during the initial stages of rolling on the primary mills. These defects may occur at any angle to the rolling direction but are most prominent in the transverse or nearly transverse direction. The appearance of ingot cracks on the rolled product varies with respect to their angle of formation and with the extent of rolling after their first occurrence. Ingot cracks are also termed "deep seams," "arrowheads," "irregular cracks," and "transverse cracks." Fig. 1 shows ingot cracks in a rolled bloom.

This report summarizes: 1—the exploratory investigation of commercial-size ingots; 2—the laboratory investigations related to determining the source of ingot cracks and developing corrective methods; and 3—the plant evaluations of laboratory methods of decreasing ingot cracks.

Exploration of Ingot Cracks in Commercial Ingots

Materials and Experimental Work: At the Duquesne Works of the United States Steel Corp., five heats of killed, fine-grained C1020 steel were selected for the exploratory phase of the seam-research program. The heats were made by open-hearth practices that are considered most conducive to good surface quality. They were top-poured into 22x25 in. big-end-up hot-topped molds. One as-cast ingot and four other ingots rolled to the following cross-sectional sizes were set aside from each heat: 16x20 in.; 9½x10 in.; 5x5 in.; and 2x2 in. The processing of the heats, from the time of charging in the open hearth furnaces to the end of the rolling operation on the primary mill, was observed carefully and recorded.

The cast ingot and the four rolled ingots from each heat were examined for surface defects. The cast ingots were split longitudinally near the vertical center plane to expose the ingot structure beneath badly cracked regions. Also, a series of transverse sections was cut at about 1 in. intervals in a badly cracked area of one ingot. All sections were ground, polished, examined by sulphur printing and deep etching, and photographed. The rolled ingots

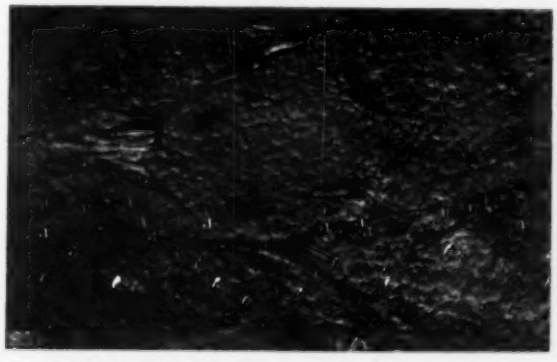

Fig. 1—Photograph of ingot cracks in a bloom section. Area reduced approximately 70 pct from actual size for reproduction.

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Discussion on this paper, TP 3775C, may be sent, 2 copies, to AIME by Oct. 1, 1954. Manuscript, Feb. 10, 1954. New York Meeting, February 1954.

The Transactions papers appearing in the Journal of Metals during 1954 will subsequently appear in AIME Transactions Volume 200, and may be permanently referenced to that volume.

Fig. 2—Vertical deep-etched section from a commercial 22x25 in. ingot that was split 2 in. off the vertical center plane. Arrows point to ingot cracks. Area reduced approximately 98 pct from actual size for reproduction.

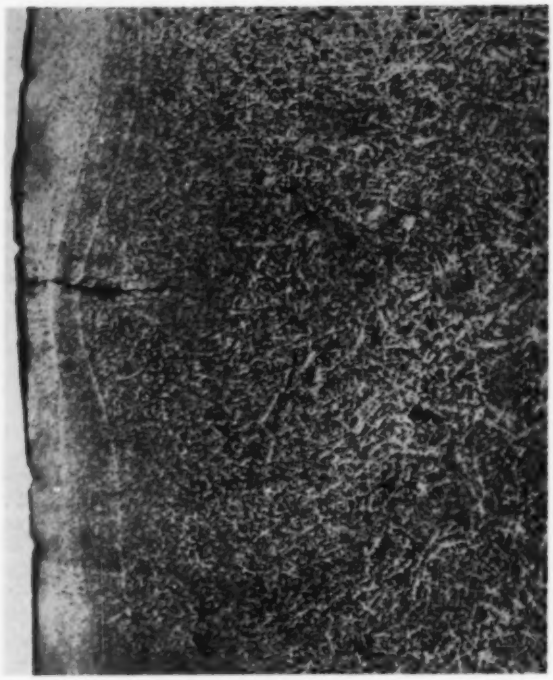


Fig. 3—Photograph of sulphur print representing an area on the vertical section shown in Fig. 2. Note the association between the ingot crack and the wavelike pattern of bands near the ingot surface. Area reduced approximately 40 pct from actual size for reproduction.

were examined by deep etching longitudinal and transverse sections through major surface defects. Some of the major defects in the cast and in the rolled ingots were examined microscopically.

Results and Discussion: Observations on the primary mill indicated that most of the ingot cracks in these heats occurred during the initial breakdown passes and that a few occurred during the later stages of rolling. The cracks were not confined to any particular position in an ingot. They occurred at random orientations, but those perpendicular to the rolling direction were by far the most prevalent. With each pass on the primary mill, the cracks gradually became re-oriented in the rolling direction. The tendency to develop ingot cracks varied widely among the heats and ingots within a heat.

The most significant and interesting phenomenon was revealed in sulphur prints and deep-etch tests of vertical sections from as-cast ingots. Near the surfaces of the ingots, between the chilled and columnar zones, were bands of lines that were more resistant to acid attack than the surrounding metal. These bands appeared in wavelike patterns. In numerous regions where the waves of bands were nearest the ingot surfaces, either cracks or intersections of dendritic crystals or both occurred. (Intersections of dendritic crystals in the columnar zone create planes of weakness from which ingot cracks may develop subsequently as a result of the stresses produced in an ingot during cooling or during rolling.) Fig. 2 illustrates the prevalence of ingot cracks in a vertical, deep-etched section from a commercial-size ingot. Figs. 3 and 4 indicate that these cracks and intersecting dendritic crystals are associated with the wavelike pattern of bands near the ingot surface. As was expected, the dendritic crystals in other regions of the columnar zone were aligned at

a fairly uniform angle to the ingot surfaces, with the result that the crystals were substantially parallel to one another. Transverse deep-etch tests also showed a wavelike pattern of bands.

Such bands of lines between the chilled and columnar zones have been observed and reported by other investigators.[1-7] The deep-etch tests of cast ingots illustrated in Humfrey's paper[1] revealed bands of lines. The white bands are not confined only to killed steels; similar patterns are visible in cast sections from rimmed steel ingots presented by Hultgren and Phragmen.[2]

Binnie[3] observed pronounced banding only in the bottom half of a medium carbon, killed-steel ingot that was bottom-poured, but was unable to detect segregation of elements across the bands. He also observed that the dendritic crystals extended through these bands. In a discussion of Binnie's paper, Northcott[4] and Fell[5] suggested that the banded effect resulted from turbulence of liquid steel during pouring.

In his paper, Northcott[6] conclusively demonstrated that turbulence of molten steel in a mold caused bands of lines. He produced a severe and extreme condition of heavy bands in a cast ingot by stirring the liquid steel in a mold with metal rods soon after pouring. On the other hand, with bottom-pouring or with top-pouring through a 16 hole tundish, only a few light bands occurred.

In discussing the bands of lines observed in cast ingots selected for their investigation, Mackenzie and Donald[7] state: "The banding effect was probably the result of a combined effect of forced convection and superheat. As the steel ran into the mold, it would set the liquid in motion. A flow of metal against the solidifying surface would check the growth of columnar crystals, and when the flow was streamlined, solidification would be inhibited as long as the liquid was superheated. If the flow was turbulent, there would be intervals during which solidification took place, alternating with periods during which solidification was inhibited, so that the primary structure presented a banded appearance."

Although bands of lines were reported by many investigators, none have associated their wavelike appearance with ingot-crack formation despite the fact that this effect is discernible in photographs of many split ingot sections appearing in publications.

In this investigation, a limited study of the bands of lines, involving macro-etching, microscopic examination, and microhardness surveys, indicated that the bands of lines were similar to centers of dendrites and that the mode of solidification of the bands was similar to that of dendrite centers. The bands of lines were not considered detrimental to steel quality, nor was their manner of formation of such importance as to warrant extensive study. They served only as an indication of an initial solidification front during the pouring operation; hence, laboratory experiments were directed accordingly.

Laboratory Experiments

Materials and Experimental Work: The laboratory phase of the seam-research program was classified into four sections.

In the first section, a study was conducted to determine whether bands of lines that were observed near the surfaces in deep-etched sections of commercial-size ingots are related to solidification fronts that occur during the pouring operation. Induction furnace heats of 60 lb were melted to C1020 composition and were top-poured into 4x4 in. big-end-up cast iron molds, Fig. 5. The heats were melted in the induction furnace and were lip-poured through the refractory-lined pouring boxes and into cast iron molds supported on trunnions. Soon after pouring, the molds were upset by rotating them on the trunnions so that only thin solidified ingot shells remained in the molds.

In the second section, experiments were conducted to determine whether solidification time, carbon content, pouring temperature, pouring rate, and pouring height affect the irregularities in thickness of an ingot shell. For this section of the laboratory study, 28 ingot shells were made.

In the third section, an investigation was conducted to develop a method for studying flow patterns during pouring. For simulating liquid steel, a dilute suspension of bentonite in water was used. A slice model of an ingot mold was made of transparent plastic material with internal dimensions of 1x5 in. and simulated a commercial 22x22 in. ingot mold. The pouring equipment for the model was designed to simulate the pressure and velocity encountered in pouring steel through a 2 in. diameter nozzle with a 10 ft height of metal in a ladle. A Mariotte bottle supplied the bentonite-water suspension at a constant pressure to a cylindrical vessel fitted with a stopper rod and a nozzle.

The manner in which the equipment was used for fluid-flow study is illustrated in Fig. 6. The bentonite suspension flowed from the Mariotte bottle to the cylindrical vessel and through the nozzle into the transparent slice model placed between polarizing and analyzing screens. A light beam, polarized by the polarizer screen, passed through the model. Interference patterns caused by movement of the bentonite suspension in the model were photo-

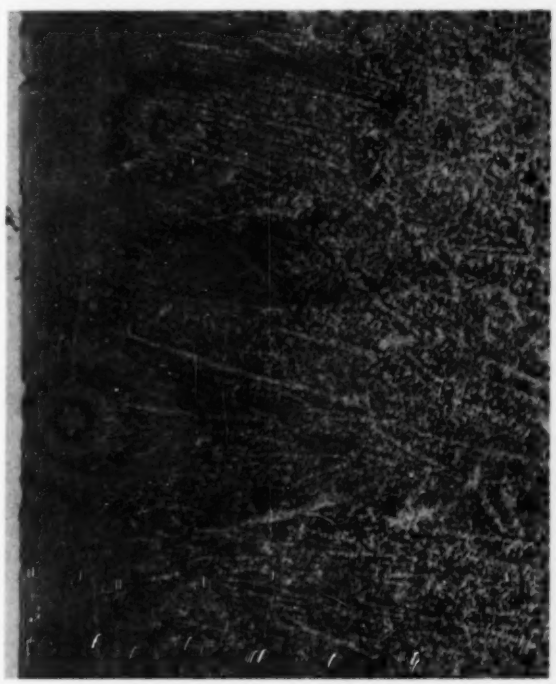


Fig. 4—Photograph of sulphur print representing an area on the vertical section shown in Fig. 2. Note the association between the ingot crack, intersecting dendritic crystals, and the wavelike pattern of bands near the ingot surface. Area reduced approximately 40 pct from actual size for reproduction.

graphed through the analyzer. Motion pictures of the following pouring conditions were taken at 64 frames per second (4 times normal speed): pouring with the stream off-center in the mold; pouring with the stream centered in the mold; and interrupted pouring, as is practiced on hot-topped ingots.

In the fourth section of the laboratory program, experiments were conducted to determine whether the pouring techniques described below would either retard solidification during pouring or control turbulence to the extent of eliminating the conditions associated with the formation of ingot cracks.

Fig. 5—Photograph of the laboratory equipment used for melting induction-furnace heats and for dumping ingots.

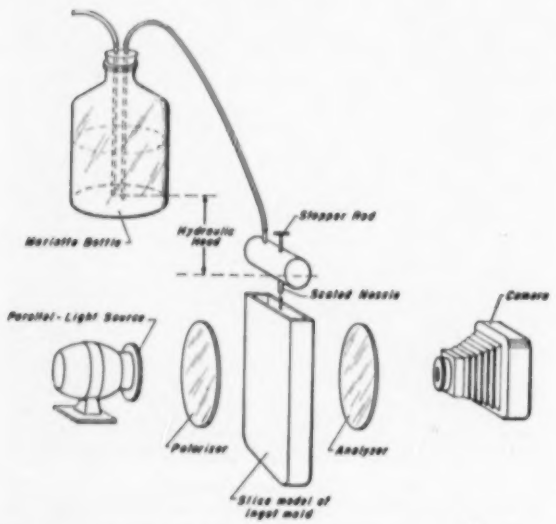


Fig. 6—Diagram illustrating equipment used in simulated ingot-pouring experiments.

As in previous tests, 60 lb induction-furnace heats were melted to C1020 composition; and immediately after pouring the heats by different procedures, the molds were dumped so as to produce ingot shells by means of the arrangement shown in Fig. 5.

One method was investigated as a means of retarding solidification rates during pouring process:

Pouring Through Molten Slag: About 3 lb of slag consisting of the following materials was placed in a graphite crucible and melted to temperatures ranging from 2400° to 2750°F: 4 parts lime, 4 parts silica, 2 parts cryolite, and 1 part fluorspar. The molten slag was poured into a cast iron mold prior to pouring the steel through a ½ in. diameter nozzle in the pouring box.

Four methods were investigated to determine whether turbulence during pouring can be controlled sufficiently to prevent the irregularities in ingot-shell thickness:

Multiple-Stream Pouring: The first method tried was pouring into a mold with several streams. The following openings were used in each pouring box for the multiple-stream-pouring experiments: four

Fig. 7—Photograph of a dumped 60 lb ingot illustrating the irregular thickness of a shell that was cast in a manner simulating commercial practice. Arrows point to cracks in the shell. Area reduced approximately 85 pct from actual size for reproduction.

⅝ in. diameter holes; twelve ¼ in. diameter holes; and four slots, each 5/8x3/16 in.

Extended-Nozzle Pouring: For the second method, a mold was placed on a platform that could be raised or lowered readily. The pouring box was fitted with a ⅜ in. diameter nozzle that extended to the bottom of the inside cavity of the mold when the platform was in the raised position. During pouring, the mold was lowered gradually so that the bottom of the nozzle was in constant contact with the rising liquid surface but did not penetrate it to a depth greater than ½ in.

Glass-Wool Packing in Molds: For the third method, glass wool was loosely packed inside the entire mold cavity, and then the steel was poured through a ½ in. diameter nozzle into the mold.

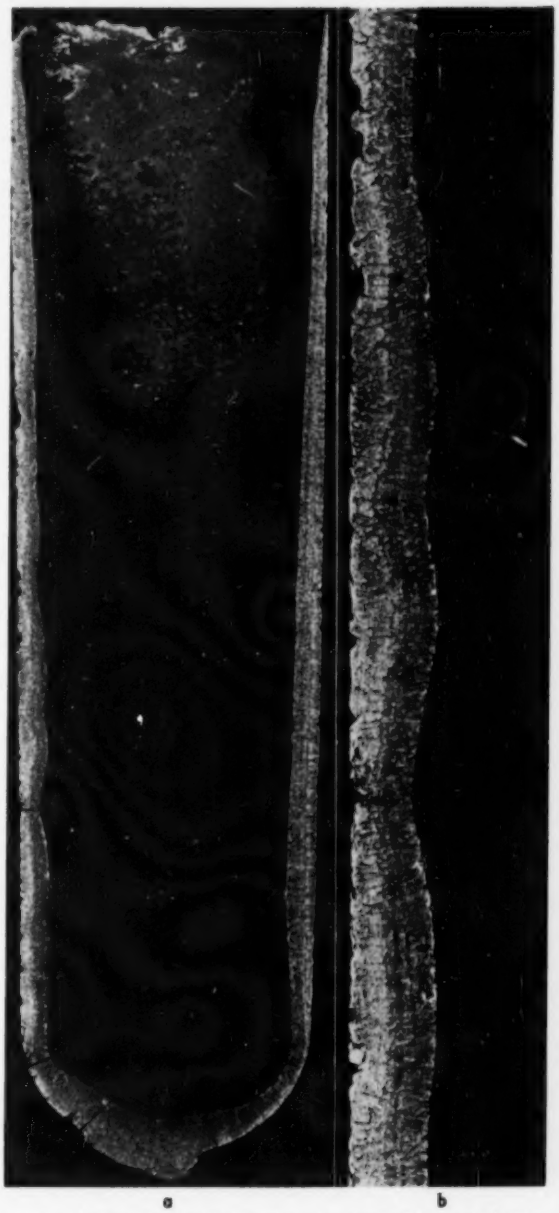
Pouring Through Stovepipes: For the fourth method, a 2 in. diameter stovepipe with a wall thickness of 1/16 in. was placed inside the mold; it extended along the entire height of the mold. Horizontal bars were welded to the top of the pipe and fastened to the top of the mold so that the stovepipe would remain rigid during pouring. The heat was poured through a ½ in. diameter nozzle in the pouring box; the stream entered the mold inside the stovepipe.

The ingot shells from all the foregoing laboratory studies were sectioned through vertical center planes and examined. Some of the shells were deep etched in hot 50 pct HCl and photographed.

Results and Discussion: The shells that were made for determining the relationship between bands of lines and initial rates of solidification varied erratically in thickness regardless of ingot position and had a wavelike contour along the interior surface in both vertical and horizontal directions, Figs. 7 and 8. These shells indicate that the conditions affecting solidification during pouring have a random effect. Deep-etch tests of longitudinal sections through the shells disclosed bands of lines similar to those found in commercial ingots. These bands of lines were approximately parallel to the wavelike contour of the ingot shells, Fig. 8. This observation indicated that the bands of lines represented a series of solidification fronts during pouring.

The exploratory plant study, together with the laboratory study, suggests the following explanation for the formation of ingot shells of erratic thickness and for subsequent occurrence of ingot cracks in regions where the shells are initially the thinnest:

As the mold is being filled, the steel adjacent to the mold walls solidifies rapidly and forms an ingot shell. Also during the pouring, the impact of the stream creates turbulence (churning or circular motion) of erratic severity in the direction of the mold walls. This turbulence below the rising surface carries superheated liquid steel to the cooling zone adjacent to the solidifying shell, and causes the shell to be uneven in thickness by momentarily retarding solidification rates in some areas more than in others and/or by unequally eroding the shell already formed. However, the direction of dendritic-crystal growth during the course of solidification after pouring is affected by the contour of the solidification front produced during pouring. Dendritic crystals grow at a fairly constant angle to a solidifying front. In regions where the solidification front is essentially in a single plane, the dendritic crystals are substantially parallel to one another. But in regions where the solidification front has a wavelike contour, the dendritic crystals intersect in the



a b

Fig. 8—Vertical deep-etched section of a dumped ingot shell illustrating the wavelike pattern of bands of lines following the contour of the interior surface of the shell. a—Area reduced approximately 70 pct from actual size for reproduction. b is magnified 3X more than a.

originally thin areas of the ingot shell. Such intersections of dendrites create planes of weakness in which cracks develop, particularly when stresses resulting from cooling or rolling occur in a direction transverse to the planes of weakness.

The solidification occurring during pouring and the turbulence resulting from the impact of the stream are considered detrimental principally because of the formation of columnar crystals. If dendritic crystals in the columnar zone of a cast ingot structure could be eliminated by some means, such as by controlling solidification rates or by inoculating the steel with special addition agents, then the two aforementioned factors would play a lesser role in the formation of ingot cracks. However,

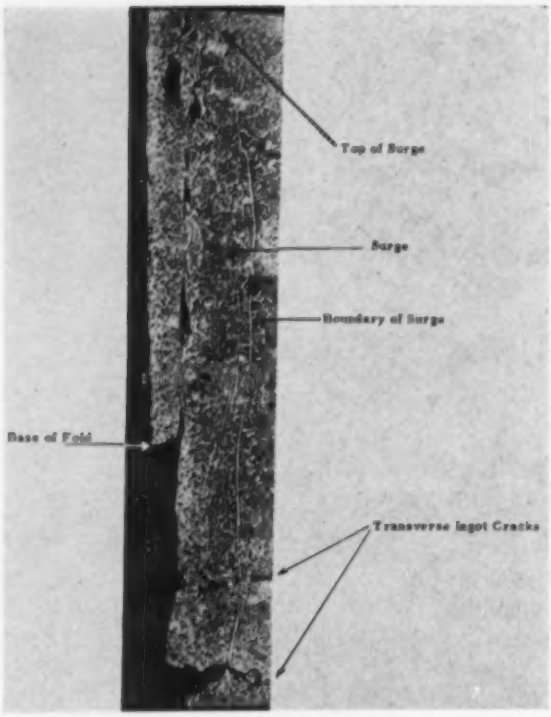


Fig. 9—Vertical section through an ingot illustrating a fold and transverse ingot cracks.

methods of eliminating or decreasing the thickness of the columnar zone were not investigated.

As was indicated in further experiments with dumped ingots, irregularities in the thickness of an ingot shell gradually disappear as the solidification time increases. By that time, however, the dendritic crystals will have intersected already and created planes of weakness.

Ingot shells of nonuniform thickness do not occur only in steels of 0.20 pct C content. This condition also occurred in laboratory ingots ranging from 0.08 to 0.45 pct in carbon content. The effects of pouring temperature, rate, and height on conditions associated with ingot cracking could not be evaluated properly because of their interrelation and because of the continual changes occurring in the characteristics of a stream when only 60 lb of metal is poured.

However, certain trends were indicated by this study. Combinations of high pouring temperatures, fast pouring rates, and considerable pouring heights develop severe irregularities in ingot-shell thickness. Low pouring temperatures and slow pouring rates develop a rough ingot surface that also may retard initial solidification rates nonuniformly by producing an insulating effect in localized areas in ingot-shell thickness. Grossmann and Stephenson[5] have shown in earlier research work that such an insulating effect produced by an ingot surface develops ingot cracks in the following manner:

During pouring, the surging and splashing of molten metal creates a wave motion and causes the metal to strike the sides of the mold above the rising surface. When the waves of molten metal recede temporarily, fins of solid metal remain above the surface. These fins, because of contraction, pull away from the mold walls and leave air spaces between the walls and the ingot surface. As the metal continues to rise, the metal flows over the fins and fills part of the air space. The space that is not filled with metal acts as an insulator in that region, retards initial solidification during pouring, and causes the dendritic crystals to intersect and to develop ingot cracks, Fig. 9.

The apparatus that was developed for simulating the pouring of steel and for studying fluid flow, adequately demonstrated the circular motion of a liquid in a mold during pouring, Figs. 10 and 11. Motion pictures of the simulated top-pouring practice showed that the flow created by the impact of a stream is extremely erratic, particularly in the vicinity of the mold walls. The flow pattern is not consistent even where the pouring stream is centered. The severity of the upward motion of the liquid along the mold walls appears to be of such a magnitude that, in pouring steel, it would momentarily retard solidification in localized areas or erode parts of the solidified ingot shells.

The objective in the last series of laboratory experiments was to control either the initial rate of solidification or the turbulence, since the simultaneous occurrence of these two factors contributed chiefly to the formation of ingot cracks.

In pouring through molten slag, the steel displaced the slag, which coated the walls above the rising metal surface. Such a coating of hot slag on the mold walls retarded initial solidification rates

Fig. 10—Photograph of a slice model illustrating simulated pouring with bentonite-water suspension as observed in transmitted polarized light. A 2 in. nozzle with a 10 ft head of metal in the ladle and a 22x22 in. ingot mold are simulated. Ingot mold half filled. Area reduced approximately 40 pct from actual size for reproduction.

Fig. 11—Photograph of a slice model illustrating simulated pouring with bentonite-water suspension as observed in transmitted polarized light. A 2 in. nozzle with a 10 ft head of metal in the ladle and a 22x22 in. ingot mold are simulated. Ignot mold three-fourths filled. Area reduced approximately 40 pct from actual size.

during pouring sufficiently to produce an ingot shell that increased uniformly in thickness from top to bottom, Fig. 12. This pouring method produced on the ingot surface a thin coating of slag, which flaked off when the ingot was stripped. In many areas, the ingot surface had a bright metallic surface beneath the slag coating.

In pouring through a pouring box with several openings, the turbulence of individual streams appeared to be offset sufficiently to produce the desired effect, since thickness irregularities in an ingot shell were eliminated.

Turbulence was decreased considerably in extended-nozzle pouring by the continuous contact of metal from the pouring box to the rising metal in the mold.

Ingot shells similar to the one shown in Fig. 12 were produced by the two foregoing methods.

Pouring into molds packed with glass wool and pouring through stovepipes were ineffective in controlling the turbulence of the metal.

Plant Evaluations

Methods and Procedures: Two laboratory methods, pouring through molten slag and multiple-stream pouring, were investigated at Duquesne Works on 22x25 in. big-end-up ingots of killed, fine-grained C1020 steel. These methods were not intended necessarily as practicable solutions to the problem; they were investigated primarily to determine whether the laboratory findings relating to the mechanism of ingot-crack formation extend to commercial-size ingots.

Pouring Through Molten Slag: A tilting-type, gas-fired furnace with a hearth capacity of 8 cu ft was constructed at the laboratory and installed on the pouring platform of No. 2 open hearth at Duquesne Works. In this furnace, slag consisting of the following materials was melted at 2250° to 2670°F: 4 parts lime, 4 parts silica, 1 part cryolite, and 1 part fluorspar. One mold from a drag of molds for each of six heats was set aside for pouring through molten slag. The slag-melting furnace was tilted, and 350 to 600 lb of molten slag was poured into this

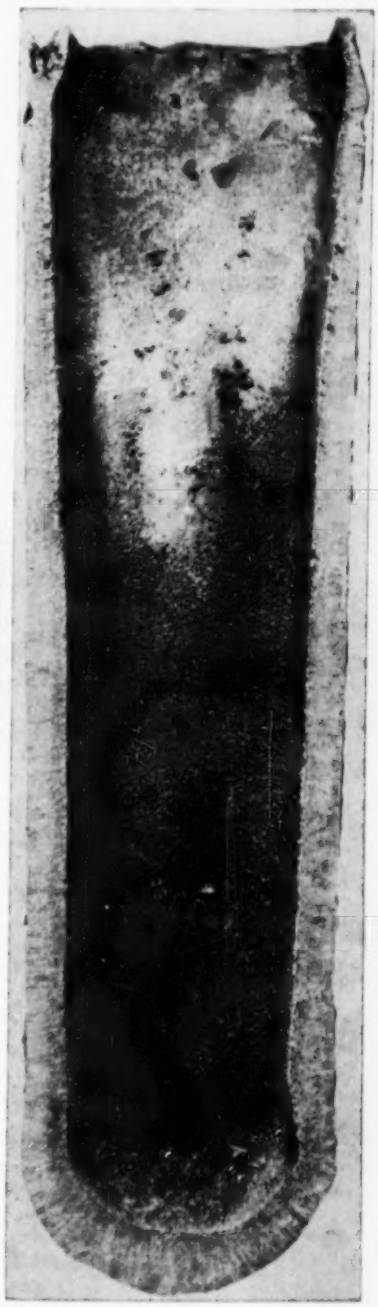


Fig. 12—Vertical deep-etched section of an ingot shell that was produced by pouring molten slag. Note the uniformly increasing thickness from top to bottom. Approximately one-half actual size.

Table I. Data on Commercial Heats Selected for Plant Experiments with Pouring Through Molten Slag

Heat No.	Temperature, °F: Tap	Pour from Ladle	Pour from Basket	Total Number of Ingots Cast: From Ladle	From Basket	Through Slag	Nozzle Diameter, In.: Ladle	Basket
100	2980	2900-2850	2865-2805	7	25	1	2½	1½
200	2980	2880-2845	2845-2800	19	15	1	2½	1½
300	3000	—	—	27	—	1	3	1¾
400	—	2910-2865	2850-2800	2	31	1	2½	1¾
500	3020	—	—	9	25	1	1¾	1½
600	3000	—	2840-2805	7	22	1	2½	1½

mold prior to casting a 9000 lb ingot. The other ingots in the six heats were cast either directly from a ladle (direct poured) or from a ladle through a tundish or basket (basket poured). Data relative to the six heats and to the slag-poured ingots are listed in Tables I and II.

The identities of the slag-poured ingots, together with the direct-poured and basket-poured ingots, were maintained throughout charging, heating, rolling, and conditioning. Except for maintaining the identities of the ingots in the three groups, the six heats were processed in the same manner as regular heats.

During rolling, all ingots of the six heats were observed carefully for ingot cracks. In the conditioning yards, the surface quality of blooms from the slag-poured ingots was further examined and compared with the surface quality of blooms from other ingots of the heats. The blooms also were examined for internal quality by deep etching transverse sections from top, middle, and bottom ingot positions.

Multiple-Stream Pouring: In the initial phase of this study, both multiple-stream pouring and slow pouring were investigated and compared with direct pouring and basket pouring. A refractory-lined pouring box with either one, two, three, or four 1 in. diameter nozzles was placed above each of two molds. Only two ingots in each of five heats were cast with such a pouring arrangement; the remainder of the steel from each heat was direct poured and/or basket poured.

Since the results of the initial study indicated that an improvement in surface quality was effected by such a procedure, the experimental work was modified so that an entire heat or any desired number of ingots might be cast with one pouring box suspended from the bottom of a ladle.

Three heats were poured through the suspended pouring box; the diameter of the nozzle in each ladle was 2½ in., whereas the diameters of the four nozzles in the pouring box were varied. From the first heat, 8 ingots were cast through four 1 in. diameter nozzles in the suspended pouring box; 12 ingots from the second heat were cast through four 1½ in. diameter nozzles; and 6 ingots from the third heat were cast through four 1½ in. diameter nozzles. All other ingots from the three heats were direct poured from the ladle. Data relevant to the conditions of multiple-stream pouring and slow pouring are shown in Tables III and IV.

As in the slag-pouring experiments, the identities of the specially poured ingots were maintained throughout processing. Also, the procedure followed in evaluating the surface quality of the two groups of ingots for the three heats was the same as that used in the slag-pouring experiments.

Results and Discussion: *Pouring Through Molten Slag:* When the six heats were rolled on the blooming mill, all the slag-poured ingots, except one that was overheated in the soaking pits, were superior to all other ingots with respect to cracking; that is, the slag-poured ingots were rolled into the desired bloom sizes without developing ingot cracks, whereas many of the regular ingots cracked so severely that it was necessary to roll them into an intermediate size for surface conditioning. Even for those ingots that were rolled directly to the desired sizes, extensive conditioning was required to remove the surface defects resulting from ingot cracks. Only light surface seams and cracks were found in the product of the slag-poured ingots.

Deep-etch tests of product from the slag-poured ingots revealed varying amounts of entrapped slag in the ingot body. Only one slag-poured ingot appeared to be free of entrapped slag. Although there were indications that this pouring method could be improved to overcome the problem of entrapped slag, the process was considered too troublesome and costly for commercial use in large tonnage shops; accordingly, additional trials were discontinued. These experiments, however, demonstrated that the laboratory findings with small ingots also applied to commercial-size ingots.

Multiple-Stream Pouring: Of the specially poured ingots in the initial trials, multiple-stream pouring with four streams was the most effective in reducing the incidence of ingot cracks. Slow pouring appeared to decrease only the severity of ingot cracks and not their frequency. These results were based on observations during rolling and on actual counting of ingot cracks in the rolled blooms.

Because multiple-stream pouring appeared promising, the experimental work was modified so that an entire heat or any desired number of ingots

Table II. Pertinent Data on the Special Slag-Poured Ingots Obtained from Commercial Heats

Heat No.	Ingots Poured	Pouring Temperature, °F: Slag	Steel	Pouring Time, Sec: Slag	Steel	Slag Poured, Lb
100	11	2250	—	—	—	350
200	16	2630	—	35	—	400
300	10	2600	—	30	—	400
400	27	2530	2860	40	—	450
500	1	2550	2950	60	120	600
600	15	2670	2930	35	120	450

Table III. Data on Slow Pouring and Multiple-Stream Pouring. Individual Boxes per Ingot

	Pouring Time for Ingot Body, Sec: 1st Heat	2nd Heat	3rd Heat	4th Heat	5th Heat
One 1 in. nozzle pouring box	375	330	Unable to pour	—	—
Two 1 in. nozzles pouring box	—	—	—	150	140
Three 1 in. nozzles pouring box	110	—	—	—	—
Four 1 in. nozzles pouring box	—	110	130	105	120
1¾ in. nozzle basket poured	100	85	105	85	105
2½ in. nozzle direct poured	40	35	40	35	40

Table IV. Data on Multiple-Stream Pouring. Suspended Pouring Box

Heat No.	Diameter of Nozzle in Ladle, In.	Diameters of 4 Nozzles in Pouring Box, In.	No. of Ingots Poured	
			Through Box	Direct
A	2½	1	8	25
B	2½	1½	12	11
C	2½	1½	6	28

could be cast with one multiple-hole pouring box suspended from the bottom of a ladle. Eight ingots from the first heat were cast through a suspended pouring box equipped with four 1 in. diameter nozzles. Observations during rolling and examination of the rolled blooms indicated that the surface quality of the multiple-stream-poured ingots was significantly superior to that of the other ingots in the heat. Because of the excessive pouring time required for the special pouring arrangement, the 1 in. diameter nozzles in the pouring box were replaced with 1½ in. diameter nozzles for the next two heats. The surface quality of these two heats was better than average for this grade. The larger diameter nozzles, however, did not effect an improvement in surface quality. Since pouring difficulties occurred in all three heats for which a pouring box was suspended from a ladle, further experiments for evaluating other-size nozzles and more than four nozzles were discontinued until improvements in the ladle-stopper riggings have been made.

Summary

A study of ingot cracks in killed, fine-grained C-1020 steel revealed that the major source of such defects is associated with conditions arising during the top-pouring practice. Controlling these conditions effects a significant improvement in surface quality. However, it must be recognized that ingot cracks may originate in other sources which were not investigated in the present program. Results of both plant and laboratory experiments may be summarized as follows:

1—*Exploratory Plant Studies:* Sulphur prints and deep-etch tests of vertical sections from commercial-size ingots revealed an interesting phenomenon associated with the formation of ingot cracks. Near the surfaces of the cast ingot sections, bands of white lines occurred in wavelike patterns; in numerous areas where the bands were nearest the ingot surfaces, cracks and/or intersecting dendrites occurred. The intersecting dendrites were considered as planes of weakness along which ingot cracks developed, especially when stresses, as those produced during rolling, occurred in a direction perpendicular or nearly perpendicular to these planes.

2—*Laboratory Studies:* The initial phase of the laboratory program demonstrated that the bands of white lines indicated a series of solidification fronts which formed during the pouring operation. Ingot shells that were produced by dumping 60 lb ingots had wavelike contours along the interior surfaces. Deep-etch tests of vertical sections from these ingot shells showed that bands of white lines similar to those observed in commercial ingots appeared in wavelike patterns and that these bands were about parallel to the contour of the interior surfaces of the ingot shells.

Results of dumped-ingot experiments and fluid-flow studies suggested an explanation for the formation of ingot cracks: As the mold is being filled, two conditions occur simultaneously, 1—the metal adjacent to the mold walls solidifies rapidly, and 2—the impact of the stream creates turbulence that moves superheated metal in the direction of the mold walls and momentarily affects the thickness of an ingot shell in localized areas. The combined effect of the two conditions produces an ingot shell that is thin in some areas and thick in others. In such a shell, stress concentration is greatest in the thin areas; consequently, cooling stresses often cause ingots to crack in these areas prior to heating and rolling. Furthermore, the contour of the solidification front affects the direction of dendritic crystal growth and causes intersections of dendrites, which, as was mentioned previously, are another source of weakness during rolling.

Slag pouring was an effective laboratory method of retarding solidification during pouring. Multiple-stream pouring and extended-nozzle pouring were effective laboratory methods of controlling turbulence.

The laboratory method of dumping ingots to produce ingot shells, described above, is considered a useful test procedure for evaluating methods of eliminating ingot cracks.

3—*Plant Evaluation of Laboratory Methods:* Pouring into molds containing molten slag is an effective method of decreasing the incidence of ingot cracks in commercial-size ingots. But this procedure is too troublesome and costly for commercial use. Multiple-stream pouring produces a significant reduction in the frequency of ingot cracks and may prove satisfactory in some open hearth and electric furnace shops for curtailing cracks.

Although the last two methods may not be considered practicable for commercial use, their effectiveness indicates that the laboratory results extend to commercial practice.

Acknowledgments

The authors gratefully acknowledge the cooperation of Messrs. R. W. Carter and W. R. Dick, Duquesne Works, United States Steel Corp., in conducting the plant studies; of Homestead District Works, United States Steel Corp., in deep etching and photographing many of the ingot shells; and of the Crucible Steel Co. of America in supplying the design for the pouring-box attachment to the ladle.

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Roasting Metallic Sulphides in a Fluid Column

by H. M. Cyr, C. W. Siller, and T. F. Steele

The development of a new metallurgical roasting device is described. It consists of a refractory column into which air is injected at various levels, forming several superimposed fluidized beds with no supporting grates. When pelleted zinc sulphide concentrates are charged, the roasted product needs no further sintering before reduction to metal.

WHEN a gas such as air is blown upward with increasing velocities through a loose mass of solid particles, marked changes in the physical behavior of the particles are noted. At first, when the velocity of the gas is insufficient to support any of the solid, the mass constitutes a "fixed bed." As the gas velocity increases until the pressure drop through the bed approaches the effective weight of the bed per unit area, the bed expands until the solid particles are supported by the air rather than by the lower particles. Some vibration of the particles becomes apparent, but little mixing occurs. This condition is called a "quiescent fluid bed."

A further increase in gas velocity imparts more separation and more motion to the individual particles until a condition of turbulence is reached. This "turbulent fluid bed" resembles a rapidly boiling liquid with the characteristic highly agitated diffuse surface and many small eruptions of the boiling mass. Different degrees of turbulence can be generated and all produce excellent mixing.

The final stage occurs when the gas velocity becomes so great as to create a "dispersed suspension." Here no surface of the mass is defined and the gas carries solid particles out of their original positions.

These changing conditions of fluidization have been studied carefully and pertinent nomenclature standardized by a committee of the American Institute of Chemical Engineers.[1]

Many mathematical analyses[2-5] have been made of the forces acting in a fluid bed. These analyses are invaluable, especially for the design of column sizes and selection of equipment. However, in a metallurgical process involving solids of many sizes with changing densities, varying temperatures, and changing gas compositions within the bed, calculations based on theory become approximate. Optimum operating conditions then are best determined experimentally.

Many applications have been made of the principles of fluid-bed action by mechanical, chemical, and metallurgical engineers. Especially when good contact between reacting solids and gases is desired, very effective results are obtained from fluid beds. They permit excellent temperature control and uniformity throughout a mass of solids in fluid action. Heat transfer to walls and any coolers is high, and fast reaction rates are attained because the solid surfaces are continuously swept clean.

The main disadvantages of fluid-bed operations are the danger of short-circuiting in a single bed, danger of incipient sintering which stops action, the necessity of avoiding large changes in particle size or density during roasting, and dust losses when particles of the charge are carried out with exit gases.

In the metallurgical field the roasting of sulphide ores to form oxides and sulphur dioxide appears to combine several operating conditions which can be achieved to advantage in a fluid bed. Roasting involves a solid-gas reaction where a high reaction rate is necessary for high capacity, where good temperature control is important in order to prevent sintering, where good heat transfer is needed, and where the density of the solids, when changing from sulphides to oxides, is not largely changed.

Short-circuiting, however, constitutes a major problem when a single fluid bed is used. Because of the turbulence of the bed, an entering particle may be in the region of the discharge before it is roasted. Hence, to attain a satisfactorily low sulphur in the calcine, a long average residence time with correspondingly low capacity is required.

The solution to this difficulty is the use of multiple stages, which in the conventional fluid-bed design requires separate hearths with feed and discharge mechanisms for each stage.

A further practical difficulty in fluid-bed roasting of flotation zinc concentrates is their fine particle size which makes a true fluid action without excessive carry-over of dust very difficult to attain, especially when the large air volumes necessary for high capacity are used.

A New Design

After considerable experimentation in the laboratory and on a semipilot-plant scale, a new method and equipment for roasting were devised which provided a unique solution to these problems. A detailed account of this development appears in the patent literature,[6] and many of the variations of this development reported herein are the subject of

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pending patent applications. The fine ore was formed into small aggregates which permitted the desired fluid-bed action with only a little dust carry-over. These aggregates were charged into the top of a "fluid column," which consisted of several superimposed fluid beds established by introducing air at several levels and thereby creating beds of varying apparent density. No grates were used, as the different stages were defined by the successive increments of injected air.

Of the many experimental columns constructed and tried, the most practical type can be illustrated by the sketch in Fig. 1. Here four zones—1, 2, 3, and 4—are shown. In the lower zone 1, just enough air was blown through ports 5 to keep the charge in an expanded state, forming a quiescent fluid bed. This air completed the roasting and swept the discharging material free of SO_2, while it also cooled the discharge and became heated itself. At inlets 6 more air was admitted. This volume added to that of the lower ports was sufficient to vigorously agitate the charge forming a turbulent fluid bed. A similar and even more violent effect occurred in the region above inlets 7. Here the combined effect of the three air injections, which had been heated and expanded to a large volume, created a vigorous suspension. The action of this section was confined by maintaining a mildly turbulent bed above it in the cone and larger diameter top. Here the lower gas velocity minimized the ejection of solids from the column.

In normal operation, the aggregates of ore were charged into the top of the column onto the top bed where they were heated to temperature and were partially roasted. As they passed into the two lower zones, roasting was vigorous and practically completed under conditions of high turbulence which minimized adhesion or sintering. The lowest bed completed the roasting and preheated the large amount of air injected through the bottom ports. The four zones maintained in the fluid column acted like four superimposed fluid beds. The charge gradually passed from one bed to the next one below, thus avoiding short-circuiting.

The unique feature of this fluid column was the absence of grates between the several stages. This obviated many mechanical difficulties connected with the construction, cleaning, and repairing of such grates. The absence of grates allowed such devices as coolers, sampling tubes, or temperature-measuring equipment to be inserted through the top and to reach any part of the interior. Charging and discharging were quick and simple.

Operation of the Roaster

The preparation of the charge was adjusted to the ore used and to the subsequent use of the material. Some zinc sulphide concentrates formed strong aggregates by simply moistening and forming pellets or flakes, then drying. Other zinc sulphide concentrates required the addition of small amounts of binder, such as sulphite liquor, bentonite, zinc sulphate, or sodium sulphate. A convenient size for flakes, pellets, strudes, or other aggregates was between the limits of 6 to 20 mesh. Smaller sizes were partially carried out with exit gases when enough air to give high capacities was used. Larger sizes created a sluggish bed, created high temperature zones, and did not rapidly roast to their center.*

* In the subsequent larger-scale development, it was found feasible to extend the size range to include some 4 mesh pellets without serious loss in capacity at a given sulphur limit.

Best fluid action was observed when the charge consisted of a range in sizes rather than of uniform-size materials.

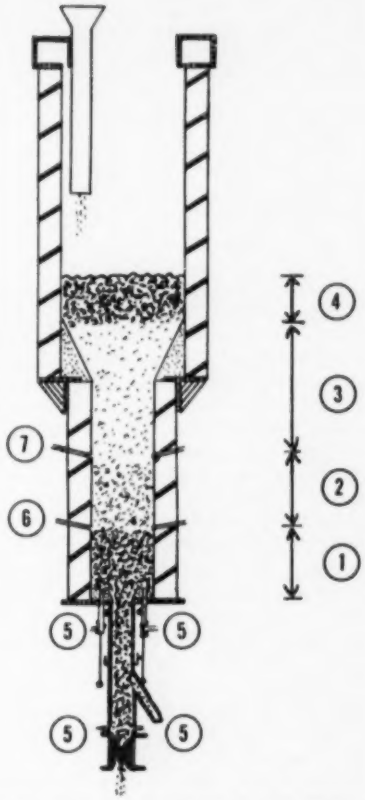

Fig. 1—General design of experimental fluid column. 1 to 4 are zones of varying fluidization. 5 to 7 are air inlets.

During roasting the dust losses from zinc sulphide concentrates aggregated into the 6 to 20 mesh size were usually in the region of 10 pct. This was influenced by the amount of binder, the mechanical densification given to the charge, the violence of the fluid action, the temperature of the roast, and the composition of the ore.

The completeness of the roasting operation could be adjusted by the amount of charge passing through the column. In most tests the sulphide sulphur remaining in the roast was about 0.1 pct. If lower sulphurs were desired, they could be reduced to 0.01 pct by using a slower feed rate.

The sulphate content of the calcine was determined largely by the lime and magnesia content of the ore, since the usual roasting temperatures were below the decomposition temperatures of these sulphates.

The temperature at which zinc sulphide concentrates were roasted normally varied from 950° to 1150°C. The temperature selected depended largely upon the sintering characteristics of the ore, which in turn were influenced by the impurity content. Best results were obtained by operating just below the sintering point. This produced rapid roasting, and any incipient sintering minimized dust losses. A temperature of 1050°C was commonly used.

The first successful fluid column developed in the laboratory consisted of a tube of 4 in. inside diameter and about 3 ft height with an expanded 9 in. top section. This size was considered to have the minimum diameter to avoid strong wall effects. Due to the large ratio of wall to volume, this column was not autogenous and was enclosed in a heated chamber in order to control temperature.

Later trials were made in a column of 12 in. inside diameter and 41 in. height plus an upper expansion chamber of 24 in. inside diameter by 51 in. high connected by a 12 in. conical section, see Fig. 2. The 12 in. fluid column was large enough to require no outside heating chamber, and the usual metallic sulphide concentrates could be roasted autogenously in it. This unit required air at 5 psi for its operation. The results obtained in this column served as guides for larger columns having much greater capacities.

Small-scale tests indicated that the capacity of a multistage fluid column was high, being of the order of at least 3 tons per day of zinc sulphide concentrates per sq ft of grate area. In the 12 in. inside diameter column with a bottom grate area of 0.78 sq ft, the normal capacity was 2 tons per day. However, if coolers were used to remove the excess heat, this capacity could be increased to 4½ tons per day. Other ways of increasing capacity by using cooling devices were also effective. One of these ways was to recirculate cooled calcine along with the fresh green aggregates. The heat-absorbing action of the inert material permitted a 20 pct increase in the feed rate into the column.

Data were secured regarding the heat transfer between roasting pellets in a fluidized column and the water in a stainless steel cooling tube. The pellets were in the −3+20 mesh size, being fed into the 12 in. fluid column continuously at the rate of 270 lb per hr, using 30 pct excess air. The pelleted green ore (Paragsha zinc concentrates) had an apparent density of 113 lb per cu ft, and the discharged calcine had an apparent density of 105 lb per cu ft. Without the water cooler, the temperature averaged 1100°C, but when 2 gal per min of water passed through the cooler, the temperature of the 500 lb bed averaged 1000°C. The cooler was a long stainless steel pipe of 13/16 in. outside diameter and 3/32 in. wall. It was immersed in the center of the charge, passing through the expanded 24 in. section and the cone section into the very active top of the 12 in. section, and thus was exposed to zones of varying charge activity which had different solids concentration. The portion of the cooler not immersed in the fluid column was well heat insulated, leaving 42 in. exposed with the area available for heat transfer amounting to 1.49 sq ft.

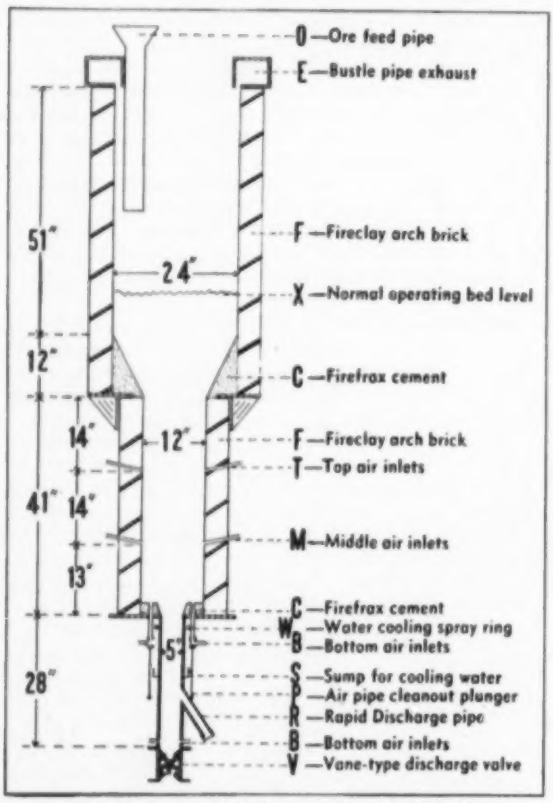

Fig. 2—Cross section of 12 in. experimental fluid column.

The average heat transfer coefficient determined from eight tests was 43.8 ±0.6 Btu per sq ft per hr per °F.

Application to Roasting of Zinc Sulphide Concentrates

Zinc sulphide concentrates from many different sources were processed in the 4 and 12 in. fluid-column roasters. These ores varied widely in composition and in particle size. Some differences were noted in roasting behavior but all were processed satisfactorily. Lowering of sintering temperature was attributed to relatively high copper and lead contents, but one ore containing 13 pct Pb was satisfactorily processed. Iron present as pyrite was observed to cause weak aggregates and increased dust losses. However, other iron compounds, such as marmatite, did not have these effects. High lime and magnesia contents were responsible for high total sulphur contents in well roasted ore. The most finely crushed ores formed the strongest aggregates, especially when a thin film of sulphate had formed on their surfaces. Since the 12 in. fluid column usually contained about 500 lb of charge during its operation, and since a feed rate of 250 lb per hr was customary, each ore aggregate underwent about a 2 hr roasting treatment.

Table I shows the data from roasting tests of four different pelleted zinc concentrates in the 12 in. fluid column, and shows a range of ores, temperatures, feed rates, air quantities, and aggregate sizes. The ore analyses are averages of the particular shipments of zinc concentrates used in the experimental work.

Ore 1 (Paragsha) was finely ground and had a high water-soluble salt content. It aggregated well without any binder, and these aggregates did not dust appreciably during roasting. Its low lime and magnesia content did not hold much sulphur. Its iron content, though high, was not in the form of pyrite.

Ore 2 (Eldona) contained considerable pyrite and low water-soluble salts, hence showed a higher dust loss in spite of the use of considerable binder. High lead and copper contents made it necessary to avoid temperatures over 1100°C for fear of clinkering during roasting.

Ore 3 (Austinville) was an easily processed material, but even a well roasted calcine still contained over 1 pct of sulphate sulphur due to its high lime and magnesia content.

Ore 4 (Eagle) contained the most pyrite of the four. It processed well, but gave some trouble from disintegration of the aggregates during roasting, resulting in dust losses often over 10 pct.

When the fluid-column roasting process for zinc sulphide flotation concentrates is compared with kiln or flash roasting followed by sintering, several advantages for the former are apparent. The step of fine grinding is not necessary for the fluid-column charge. Aggregation of the charge by flaking or

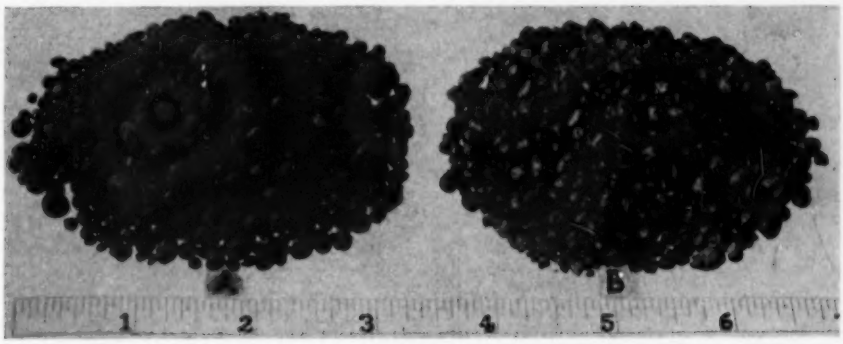

Fig. 3—Calcine from two zinc concentrates, A and B.

pelleting is necessary before fluid-column roasting but is also necessary before sintering. Because the ore is aggregated as the first step and is discharged as hard pellets, dust losses are very low. The fluid column in one operation produces a roasted aggregate suitable for subsequent reduction, whereas the other roasting method needs the second step of sintering. The fluid column device is a compact high capacity unit with no moving parts and is of simple construction.

Application to Roasting of Iron Sulphides

In addition to zinc sulphide concentrates from many sources, other metallic sulphides were successfully roasted in these columns. Pyrrhotite (FeS) can be obtained in both the massive form and as flotation fines. A massive form which was crushed through 4-mesh could be well roasted by charging into the top of the furnace in a manner similar to that used for zinc sulphide. Most of the fines smaller than 35-mesh were carried out of the column by the exit gases. However, they were well roasted and easily separated by means of cyclones and dust catchers. It was found that, when the crushed pyrrhotite containing many fines was introduced considerably below the top surface of the bed rather than on the top, lower sulphur contents were found in the dusts separated from the exit gases.

When the raw material consisted entirely of fine pyrrhotite or fine pyrite, two methods were used to roast them in the fluid column. In the first method the fines were aggregated into flakes or pellets. As was the case with zinc sulphide concentrates, some ores could be formed readily into strong aggregates using only water, whereas other ores required binders such as sulphite liquor, bentonite, borax, or sulphates. These aggregates could be roasted in the normal manner, producing a strong sulphur dioxide gas carrying little dust from the top of the column and discharging from the bottom strong aggregates of iron oxide containing low sulphur.

The second method consisted of injecting the fine unaggregated charge into a fluidizing charge of some inert material, preferably pellets of iron oxide. The injection was made by means of a tube through the side of the column near the bottom. As the fine material worked its way up with the ascending gases, roasting took place, maintaining the column at the desired temperature. The roasted dust was mostly carried out of the column by the gases. It was collected in dust catchers and cyclones and was found to be well roasted.

The concentration of sulphur dioxide in the exit gases from a sulphide-roasting operation can be relatively high. When low sulphide sulphur is desired in the calcine, it is advisable to use 10 to 20 pct excess air. Theoretically, when 20 pct excess air is used and when zinc sulphide concentrates are being roasted, a sulphur dioxide concentration of 12.3 pct SO_2 should be found in the exit gases. When pyrrhotite is being roasted, this figure becomes 10.8 pct. When pyrite is the charge, an SO_2 content of 13.3 pct can be expected, assuming complete oxidation of the iron to the ferric condition. Actually, SO_2 concentrations close to these theoretical values have been found in gas samples taken at the top of the column. However, when the exit gases are drawn by fans through much piping, lower values usually are caused by inevitable false air also being drawn into the roaster system.

Experimental work has demonstrated the applicability of this new fluid-column method to other operations involving solid-gas contacts.

Two main potential advantages inherent in the fluid-column roasting method as developed in the laboratory were believed to justify an attempt to apply it to the commercial roasting of zinc sulphide concentrates. These two advantages were:

1—The calcine would be obtained in an agglomerated form which would make possible the elimination of the conventional sintering step in certain smelting operations. With the exception of sinter roasting, processes used in the past for roasting zinc sulphide concentrates have required a second step of sintering to prepare the calcine for use in a pyrometallurgical process. While this sintering step is relied on sometimes for the final elimination of sul-

Table I. Roasting Tests of ZnS Concentrates in the 12 in. Fluid Column

	Ore 1, Paragsha	Ore 2, Eldona	Ore 3, Austinville	Ore 4, Eagle
Screen size	−3+20	−3+20	−6+20	−6+20
Binder*	None	1+2*	1+1*	1+1*
Density: lb per cu ft	114	113	120	122
Lb per hr—feed	270	228	253	219
Air x theory	1.2	1.4	1.1	1.1
Temperature, °C	1075	1075	1000	1075
Dust loss, pct	6	13	5	12
Sulphide S	0.03	0.5	0.02	0.1
Total S	0.52	1.2	1.4	0.7
Typical Analyses, Pct				
Zn	47.8	46.4	59.4	48.7
Pb	2.6	2.6	0.9	0.9
Fe	11.5	10.6†	2.4	13.1†
Cd	0.15	0.1	0.11	0.24
Cu	0.12	1.9	0.03	0.73
S	31.8	33.5	30.9	33.9
CaO	0.09	—	1.5	0.06
MgO	0.05	—	1.2	0.15
Water-soluble salts	4.8	low	0.6	0.5

* 1+2 indicates 1 pct bentonite plus 2 pct sulphite liquor.
† Fe present largely as pyrite.

phur, its essential purpose is to change the physical condition of the calcine to yield a more easily reduced charge for the smelting operation and for better zinc recovery.

2—The amount of dust to be handled in the roaster-gas recovery system and dusting losses would both be small. As contrasted with processes which do not employ an agglomerated charge, the fluid-column process operates with a material which is in essentially a nondusting form throughout the charge preparation, roasting, and calcine-handling stage. As a result, dust losses in the entire roasting operation are reduced to a minimum, and only a small percentage of the product leaves the roaster as dust.

Realization of these advantages would result in savings in plant investment and operating costs, as well as in better overall recovery of zinc.

Pilot Plant Operation

With these advantages in mind, a pilot plant with a capacity of 10 to 15 tons of concentrate per day was built to study the commercial aspects of roasting agglomerated zinc concentrates in a fluid column. The roaster operated well, producing an agglomerated calcine low in sulphide sulphur and with only a small amount of dust carried over with the roaster gas. Even at relatively high temperature, clinkering was not a problem.

At the conclusion of the pilot-plant work, it was decided to test the process on an experimental unit of commercial size to verify the pilot plant results and to obtain more accurate data for future design.

The plant has operated for about a year with good recovery of dried on-size agglomerates (−4+20 mesh) and smooth operation of the fluid-column roaster with sulphide sulphur analyses of the calcines comparable to those of calcines produced by flash roasting and sintering.

Fig. 3 shows the pellets discharged from the commercial fluid-column roaster charged with two different zinc concentrates. These pellets were hard, nondusting, and free flowing. They showed only moderate moisture pickup or decrepitation when stored outdoors.

Full commercial-scale tests of the agglomerate calcine on one battery of vertical retort smelting furnaces showed good mix-preparation characteristics, good briquetting operation, good green, coked and residue briquet quality, and metallurgical performance equal to sinter prepared by flash roasting and sintering. Tests of the agglomerate calcine on a 6000 kw Sterling Furnace compared well with those employing a sintered charge prepared by flash roasting and sintering, showing normal behavior of the charge banks, smooth smelting operation, and high condenser efficiency.

The capacity of the experimental roaster with a hearth area of 20 sq ft is 75 tons of concentrate per day for an average concentrate. Indications from the work are that the dimensions of the roaster can be increased with no loss in metallurgical performance. Tests are now under way on a larger unit.

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Technical Note

An Fe-Cr-Mo-Ni Sigma Phase

by A. G. Allten

EXAMINATION by metallographic and X-ray diffraction means of an austenitic steel containing 0.06 pct C, 1.26 pct Mn, 0.38 pct Si, 21.15 pct Ni, 18.72 pct Cr, 3.07 pct W, and 9.14 pct Mo indicated that a σ phase, which had not been reported in the literature, existed in this steel. Both the σ and carbides of the M_6C type were present in the as-forged steel. The carbides dissolved completely in the austenite at approximately 2200°F (1205°C). The σ, however, was present at all temperatures up to the solidus temperature of the steel at approximately 2375°F (1300°C). Ferrite was not found in any condition of the steel.

The faintness of some of the σ lines in X-ray patterns obtained from samples of the steel made it desirable to isolate the σ prior to verification of its entire pattern. It was found that the σ phase could be separated from samples of the steel which had been water quenched from 2250°F (1230°C) by dis-

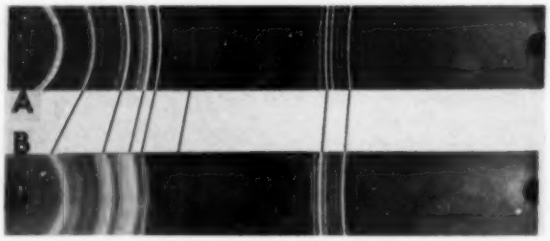

Fig. 1—Comparisons of σ phase diffraction patterns. Joined lines illustrate similarity of structures, Cr Kα radiation. a—38 Fe, 30 Cr, 16 Mo, and 16 Ni. b—Fe-Cr σ.

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solving the austenitic matrix in boiling aqua regia of the following composition: 30 parts concentrated HCl, 10 parts concentrated HNO_3, and 5 parts concentrated H_3PO_4. The H_3PO_4 prevented precipitation of tungsten and molybdenum compounds from the aqua-regia solution near the end of the extraction.

The σ was obtained from the aqua-regia extraction as a grey powder which was free of austenite and carbides as indicated by X-ray diffraction means. The composition of the σ was deduced from chemical analyses of the extracted residues as given in Table I.

The diffraction pattern of the Fe-Cr-Mo-Ni σ was compared with a pattern obtained from Fe-Cr σ. The patterns are shown in Fig. 1a and b. It was found, as shown in Table II, that all but a few very weak lines of the pattern of the Fe-Cr-Mo-Ni σ of Fig. 1a could be matched to corresponding lines in the Fe-Cr σ pattern of Fig. 1b. The interplanar spacings of the Fe-Cr-Mo-Ni σ are 1.018 times those of Fe-Cr σ.

Sully[1] has suggested that the σ phases are electron compounds. Following this suggestion, Bloom and Grant[2] and also Greenfield and Beck[3] have worked out empirical relations between atomic compositions and electron vacancies for various σ phases.

Bloom and Grant[2] assumed that the σ-phase composition is characterized by a constant number N_v of $3d + 4s$ electrons per atom calculated[2,3] as follows:

$$N_v = 5\,(V) + 6\,(Cr) + 6\,(Mo) + 7\,(Mn) + 8\,(Fe) + 9\,(Co) + 10\,(Ni).$$

The chemical symbols represent the atomic fraction of the corresponding element in the σ phase.

An average N_v, calculated by Greenfield and Beck[3] from the mean compositions of nine binary σ phases, is 6.93. Bloom and Grant[2] found an N_v value of 7.4 for the ternary 37 pct Cr, 27 pct Mo, and 36 pct Ni σ phase.

Greenfield and Beck[3] have proposed the following correlation of composition with electron vacancies N_v per atom in the 3d level:

$$N_v = 4.66\,(Mo + Cr + V) + 3.2\,(Mn) + 2.2\,(Fe) + 1.71\,(Co) + 1.6\,(Ni).$$

This relation gives an average N_v value of 3.41 for binary σ phases.

If the composition 37.6 pct Fe, 30.3 pct Cr, 16.0 pct Mo, and 16.1 pct Ni is inserted in the above equations, values of $N_v = 7.4$ and $N_v = 3.3$ are obtained. It must be remembered that the composition of the Fe-Cr-Mo-Ni σ phase, given above, is probably not the mean composition. However, the correlations are sufficiently good to indicate that the quaternary Fe-Cr-Mo-Ni σ phase obeys these empirical rules which were derived from compositions of binary and ternary σ phases.

A 15 lb induction-melted ingot was made up to the composition of the Fe-Cr-Mo-Ni σ phase. This ingot analyzed as follows: 0.05 pct C, 0.86 pct Mn, 0.45 pct Si, 15.20 pct Ni, 24.67 pct Cr, 24.45 pct Mo, and 0.07 pct N.

Table I. Composition of Sigma

	Fe	Cr	Mo	Ni
Atomic pct	37.6	30.3	16.0	16.1
Wt pct	34.4	25.3	25.0	15.3

Table II. Comparison of Interplanar d* Spacings of 38 Fe, 30 Cr, 16 Mo, and 16 Ni Sigma Phase and Fe-Cr Sigma Phase

Fe-Cr-Mo-Ni σ Phase			Fe-Cr** σ Phase	
d	I*	d/1.018	d	I*
2.61	*VW*	2.56	—	—
2.53	*VW*	2.49	2.47	*VW*
2.47	*VW*	2.43	—	—
2.41	*MW*	2.37	2.36	*VW*
2.32	*M*	2.28	2.27	*MW*
2.23	*VVW*	2.19	2.17	*VW*
2.16	*S*	2.12	2.13	*S*
2.10	*M*	2.06	2.06	*M*
2.08	*VVW*	2.04	—	—
2.06	*MS*	2.02	2.03	*MS*
2.01	*S*	1.98	1.97	*S*
1.98	*VVW*	1.95	—	—
1.96	*VS*	1.93	1.93	*VS*
1.94	*VVW*	1.91	—	—
1.92	*S*	1.89	1.89	*S*
1.87	*M*	1.84	1.84	*W*
1.79	*M*	1.76	1.76	*MW*
1.69	*VVW*	1.66	1.67	*VVW*
1.67	*VVW*	1.64	1.63	*VVW*
1.64	*VVW*	1.61	1.61	*VVW*
1.62	*VVW*	1.59	—	—
1.585	*VVW*	1.555	—	—
1.416	*M*	1.390	1.390	*VW*
1.400	*M*	1.375	1.375	*VW*
1.357	*MW*	1.332	—	—
1.350	*MS*	1.325	1.325	*MW*
1.336	*M*	1.312	—	—
1.278	*S*	1.254	1.256	*MS*
1.264	*M*	1.241	1.242	*M*
1.260	*VS*	1.238	1.237	*S*
1.254	*M*	1.231	1.232	*M*
1.247	*MS*	1.225	1.226	*MS*
1.241	*M*	1.220	1.219	*M*
1.227	*S*	1.205	1.206	*S*
—	—	—	1.197	*M*
1.210	*M*	1.188	—	—
1.206	*MW*	1.185	1.185	*M*
1.197	*M*	1.176	1.17	*M*
1.187	*S*	1.166	1.16	*S*
1.171	*VW*	1.150	—	—
1.162	*S*	1.140	Cut off	—
1.156	*W*	1.135	Cut off	—

* All d values are in the Angström units. All intensity *I* estimates are in the usual abbreviations: *VS* is very strong, *S* is strong, *MS* is medium strong, *W* is weak, and *VVW* is very very weak.

** Fe-Cr pattern was obtained from a 45 pct Cr, 3 pct Si, and 52 pct Fe alloy which was heated 1400°F 16 hr in air, powdered, and annealed 1400°F 32 hr in vacuo.

Metallographic and X-ray diffraction examination of the ingot showed that it consisted of the σ phase of Fig. 1a, in which there was dispersed a eutectic of austenite and the Fe-Cr-Mo-Ni σ. Homogenization at 2000°F (1095°C) apparently converted the austenite to σ, since no austenite lines could be found in the diffraction patterns of samples which were so homogenized.

In the as-cast and as-homogenized state, the ingot of the Fe-Cr-Mo-Ni σ was very brittle and numerous cracks could be detected in it by macro and microscopic examination. Microhardness values equivalent to 1100 VPH were obtained.

The equilibrium stability of the Fe-Cr-Mo-Ni σ phase was not established. However, no evidence was found of decomposition of the σ, and apparently it can be retained quite readily from the solidus to room temperature. This suggests that it may be possible to grow a single crystal of the Fe-Cr-Mo-Ni σ from the melt for crystallographic structure determinations.

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Effect of Aluminum on the Low Temperature Properties Of Relatively High Purity Ferrite

by H. T. Green and R. M. Brick

True stress-strain data on alloys of pure iron with up to 2.4 pct Al were obtained in the temperature range +100° to −185°C. Aluminum was found to reduce yield and flow stresses of iron at low temperatures but to have little or no effect on ductility. The effects of temperature and composition on strain hardening are discussed.

SEVERAL independent studies of the behavior of high purity iron binary alloys at low temperatures are now in progress in attempts to evaluate systematically the variables affecting the low temperature brittleness of ferritic steels. This paper reports the results of one such investigation in which the tensile properties of aluminum and aluminum plus silicon ferrites were measured from 100° to −192°C. True stress-natural strain data have been obtained in order to evaluate as many as possible of the parameters which describe the behavior of the materials involved.

In comparable studies at the National Physical Laboratory in England, iron and iron alloys of high purity have been produced[1] and tested at subatmospheric temperatures.[2] True stress-natural strain curves were obtained there also. The purest iron contained 0.0025 pct C and 0.001 pct O and N. Even this, as normalized at 950°C following hot rolling, showed little ductility at −196°C. The grain size was ASTM No. 3, and the room-temperature yield strength was 17,800 psi (which seems too high for pure iron). Some of the NPL irons contained considerably more oxygen and demonstrated intergranular fracture at −196°C. The authors[2] carefully differentiated between intergranular fractures associated with excessive oxygen content and transcrystalline cleavage with little ductility encountered at −196°C in the purer material. The cleavage stress was half again as great as that associated with intergranular fracture.

Test Material, Preparation, and Procedures

Of a number of Fe-Al alloys produced, eight were considered to be sufficiently pure for testing. Partial chemical analyses (Table I), low observed yield points, and high ductilities indicate these alloys to be comparatively pure for vacuum-melted irons of sizable ingots, 5 lb or more.

To produce the binary Fe-Al alloys, electrolytic iron was melted in air, cast into slabs, and rolled to strips 0.010 in. thick. These strips, joined into a continuous ribbon and wound into 2½ in. diameter spools, were subjected for four weeks to a moving atmosphere of purified dry hydrogen in a stainless-steel tube at 1050° to 1150°C. Charges of these spools were melted in beryllia crucibles under good vacuums (1 micron), and aluminum (99.97 pct Al) was added to the melts. Compositions of these alloys are recorded in Table I.

The ingots were hot forged and then cold rolled at least 65 pct to ⅜ in. rods which were vacuum annealed to the desired grain size, approximately ASTM No. 4, prior to machining into tensile test bars. All tensile specimens had gage sections 1 in. long, with a fillet of 1.5 in. radius to the shoulder. Gage diameters were 0.250 in. except for a few rods where additional cold work required use of a 0.200 in. gage section.

After machining, 0.002 in. was removed from the gage diameter using 240, 400, and 600-grit metallographic papers. The final polish with 600 grit left the fine scratches running in the longitudinal direction. By this means, surface metal strained during machining was removed. A few specimens heat treated after machining were similarly reduced 0.004 in. to remove any material affected chemically by the atmosphere during heat treatments, as is discussed in a later section.

Tensile tests of the eight alloys at constant temperatures from +100° to −185°C were performed in apparatus which has been described.[3] The essentials include a double-walled insulated metal vessel which contained the liquid heat-transfer medium surrounding the test specimen. A constant temperature was maintained by means of a pyrometer which regulated the pressure of dry air driving liquid air through a copper coil. Temperature variation was less than ±2°C during a specific test.

For axial straining, two lengths of case-hardened chain, terminating in simple shackles, loaded the specimen through threaded grips. The lower grip bar passed through a hole in the bottom of the test vessel to which it was joined by a thin-walled

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Table I. Compositions of the Aluminum and Aluminum-Silicon Ferrites

Alloy	Al, Pct	Si, Pct	Mn,* Pct	Ni,* Pct	Cr,* Pct	S, Pct	C, Pct	O,† Pct	N,† Pct	H,† Pct	Al_2O_3, Pct
27	0.02	0.002	0.002	0.000	0.000	0.002	—	—	—	—	0.002
26	0.20	0.001	0.002	0.000	0.000	0.003	—	0.000(9)	0.000(4)	0.000(5)	0.005
28	2.41	0.005	0.002	0.000	0.000	0.002	—	—	—	—	0.003
25(oxides)	0.20	0.002	0.002	—	—	—	—	—	—	—	0.084
11	0.012	0.16	—	—	—	—	—	—	—	—	0.009
5	0.27	0.09	0.003	<0.01	0.003	0.006	0.006	—	—	—	0.008
9	0.41	0.20	—	—	—	—	—	—	—	—	0.013
8	1.41	0.16	—	—	—	—	—	—	—	—	0.015

* Gravimetric or volumetric analyses reported. Spectrographic analyses for these elements in comparable irons prepared from the same raw materials by similar methods have reported values in the range of 0.0003 to 0.0008 pct.
† Analyses by National Bureau of Standards, courtesy of Dr. J. G. Thompson.

(0.004 in.) flexible metal bellows serving as a seal.

The simultaneous values of load and minimum diameter throughout the tensile tests were recorded autographically, using a special diameter gage. This consists of two stainless-steel arms, extending down into the test vessel and terminating with hardened stainless-steel anvils which contact the reduced section of the specimen. The movable arm actuates a microformer. As the position of the core is changed by decrease in specimen diameter, this change is matched and magnified in an autographic stress-strain recorder. Load vs diameter plots are thus produced at a nominal diameter magnification of 100:1. A Baldwin strain pacer in the circuit enabled the minimum diameter to be followed readily throughout the test. The precision of the diameter gage appeared to be 0.001 in., which was satisfactory, since elastic moduli values were not desired.

Initial and fracture diameters were measured at room temperature and are expressed as room-temperature values without correction for thermal expansion or contraction to the test temperature. Frequently, the specimen fracture was somewhat elliptical. Less often, some ellipticity was found to exist at the gage marks. In these cases, the major and minor diameters of the ellipses were recorded. Very light surface marks, from traversing with the gage anvils, were visible on the gage section of each specimen and identified the direction of strain measurement with respect to the ellipse axes.

For the calculation of the true stress-natural strain diagram, 20 or 30 points were selected on the load-diameter chart, to each of which was appended the corrected load value and the calibrated diameter. The calibrated diameter values, representing the true diameter measured during the test, were then corrected for ellipticity when necessary.

For each test the true average stress-natural strain diagram was constructed through the selected points and the following data recorded: yield stress and yield type; flow stress at strains of 0.1, 0.2, 0.3, 0.5, and 1.0; fracture stress; uniform strain; total strain; and slope of the diagram from the limit of uniform straining to a strain of 1.0.

The grain size of each specimen was determined metallographically after testing. Not all specimens had the same grain size across the entire section; both mixed and duplex grain distributions were encountered. The test data indicated agreement with the qualitative observation of Edwards and Pfeil[4] that, for the same average grain size, the nonuniform distribution produced higher strengths, therefore, reflecting more the presence of the smaller grains.

Results

The test results for three binary aluminum ferrites, 0.02, 0.2, and 2.41 pct Al, will be presented and discussed first. Attention will be restricted to specimens which were furnace cooled after recrystallizing to grain sizes ASTM Nos. 3 to 5, and which were tested at a constant rate of crosshead motion of the testing machine. To these results will be appended results of partial investigations of the effects of silicon, heat treatment, and strain rate.

Fig. 1 shows representative stress-strain curves for the 0.2 pct Al alloy at several temperatures. While only one of these particular curves shows a point between a strain of 2.0 and fracture, the upward curvature is sufficiently established by many other tests to be qualitatively accurate. It will be observed that a fairly straight-lined portion exists in these curves from just after the limit of uniform strain to strains in excess of 1.0. The slope of this portion, the "strain-hardening modulus," μ, has been measured from the limit of uniform strain to a strain of 1.0, see Fig. 1.

The changes in specified values of yield or flow stress and strain with decreasing temperature are shown in Figs. 2 to 4 for the plain aluminum ferrites. Superposition of these yield or flow-stress curves shows, particularly at a strain of 0.2, a more rapid rate of increase of stress with decrease in temperature by the 0.02 and 0.2 pct Al alloys than by the 2.41 pct Al alloy. Thus a higher aluminum content apparently reduces the yield or flow stress for a given strain at low temperatures, an effect which will be called "solid-solution softening." When the flow stress at a strain of 0.2 is plotted against alumi-

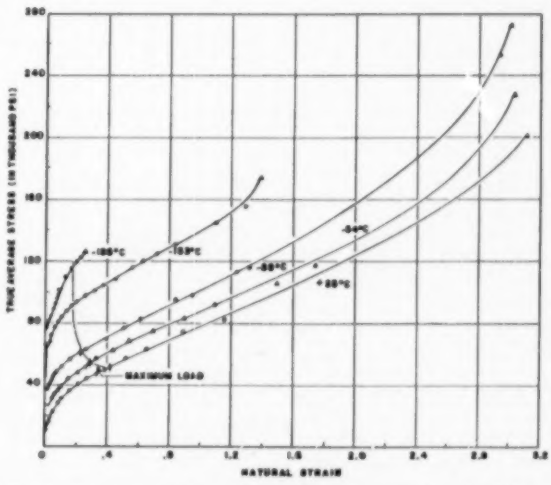

Fig. 1—True average-stress vs natural-strain curves for the 0.2 pct Al ferrite with a grain size of ASTM No. 4.

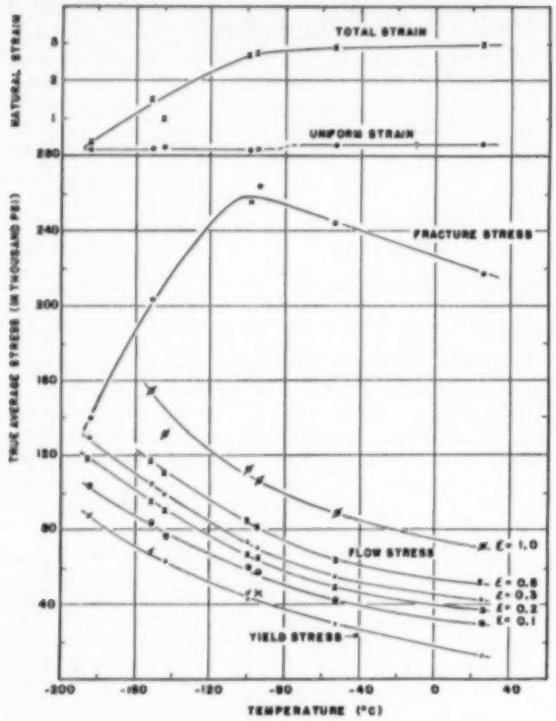

Fig. 2—Effect of temperature upon the yield stress, flow stresses at selected strains, and fracture stress (bottom) and ductility in terms of uniform and total strain (top) of the 0.02 pct Al ferrite with a grain size of ASTM No. 3 to 4 (alloy 27).

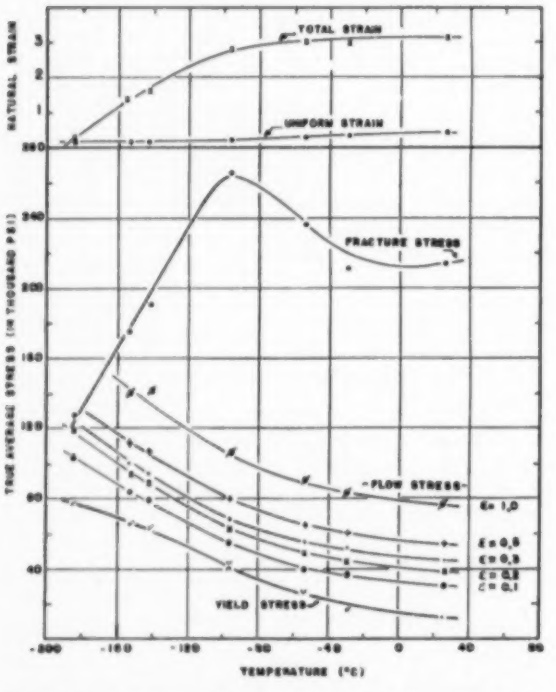

Fig. 3—Effect of temperature upon the yield stress, flow stresses at selected strains, and fracture stress (bottom) and ductility in terms of uniform and total strain (top) of the 0.20 pct Al ferrite with a grain size of ASTM No. 3 to 4 (alloy 26).

num content at various temperatures from atmospheric to −185°C, Fig. 5, the anomalous "solution softening" at temperatures below −145°C becomes manifest. The same reversal in effect of aluminum content upon the flow stress between +25° and −185°C is demonstrated at several strains in Fig. 6.

Lower total strains are associated with the higher aluminum content of the 2.41 pct Al alloy in the vicinity of room temperature. The decrease in total strain with decreasing temperature, however, is less rapid (Fig. 4), and near −185°C the difference between low aluminum and higher aluminum ferrites disappears, as is seen in the illustration.

It will be noted that these materials demonstrated great ductility above −100°C, e.g., reductions of area of 90 to 96 pct.

As the test temperature was lowered to −100°C, fracture stress increased even though total strain was constant in the 0.02 and 0.2 pct Al ferrites, or decreased gradually in the 2.4 pct Al alloy. This increase in fracture stress followed the increase of all stress values at given strains with decreasing temperature down to about −100°C. Below −100°C, the observed decrease in total strain resulted in less total strain hardening and thereby caused the fracture stress to decrease.

Discussion of Results

Fracture strain and the related fracture stress are very structure sensitive at the temperature of liquid air where the difference between flow stress and fracture stress in these alloys is small. For example, specimens of an alloy 0.20 pct Al-0.09 pct Si, held over from a group tested two years previously,[5] showed very little ductility at −185°C, whereas a specimen of the same alloy rerolled from one shoulder of a large bar, annealed to its previous grain size, and machined after annealing, had a total strain of 0.8 at liquid air temperature. The original specimens were found to have suffered subsurface oxidation during vacuum annealing which produced a network of grain boundary inclusions in the surface zone. These inclusions severely reduced low temperature ductility.

Observed differences in grain size accounted for differences in properties in a few cases. Compositional differences among specimens of the same alloy might well have existed, since the melts were slowly solidified from the bottom, the ingots were quarter-sectioned (in the cases of the aluminum ferrites), and little hot working was performed. Generally, however, differences between duplicates were small compared to the effect of temperature and composition.

The room-temperature test data show the expected increase in tensile properties with increase in aluminum content. Comparison among the aluminum ferrites of this study, those of Lacy and Gensamer,[4] and Yensen and Gatward,[7] reveals that all three sets of data show generally a progressive increase in yield and ultimate strengths with increasing aluminum content, Table II. The low yield stresses obtained by Lacy and Gensamer may be explained by their wet-hydrogen annealing.

When the logarithm of the stress at a specified strain is plotted against the reciprocal of the test temperature in degrees Kelvin, Fig. 7, two straight lines are obtained. This suggests that the temperature dependence of the flow stress is related to an "activation energy." The flow stress apparently is inversely related to the rate of some deformation

Table II. Comparative Engineering Tensile-Strength Data for Aluminum Ferrites

Alloy	Al, Pct	Room Temperature Yield Strength, 1000 Psi	Room Temperature Ultimate Strength, 1000 Psi	Grain Size
27	0.02	13.7	32	4 to 5
Yensen and Gatward	0.09	13.7	33.7	—
26	0.20	12.3	34.1	3 to 4
Lacy and Gensamer	0.50	9.0	36.6	4
Lacy and Gensamer	1.00	12.5	40.5	5 to 6
Lacy and Gensamer	1.50	14.9	42.2	5
28	2.41	25.4	46.7	3
Yensen and Gatward	2.67	30.1	49.4	—
Yensen and Gatward	5.2	45.0	61.6	—

"reaction." In the expression, $\sigma = ke^{q/RT}$, the exponent is positive because decreasing temperature results in increasing stress. It is improbable that a simple inverse relation exists between the velocity of a deformation reaction and the flow stress at an arbitrary rate of deformation. Therefore, q and k are not presented as the numerical equals of A and E in the usual expression for reaction velocity.

Nevertheless, the linearity of log σ vs $1/T$ is not peculiar to the present tests[8] and an analogy appears to exist. The slope of the line of log σ vs $1/T$ must be related to an activation energy; the steeper the line, the greater the temperature dependency of the process, and, therefore, the greater the activation energy. The value of k, in the expression for activated deformation, is the inverse analogous counterpart of a measure of the probability of, or loosely, frequency of, instances of possible reaction. This value is equal to the stress intercept at $1/T = 0$.

The activation energy for deformation, q, can be considered to be the energy necessary to initiate movement of dislocations, and the value, k, a measure of the dislocations available for deformation. In Fig. 7, for instance, it will be noted that in the temperature range +100° to −100°C the slopes of the log σ vs $1/T$ curves for flow at $\epsilon = 0.2$ and 0.5 indicate a value for q in the vicinity of 0.4 kcal per mol, while the slopes for the yield stresses indicate values for q in the vicinity of 1.2 kcal per mol. This suggests a lesser activation energy for continuing the movement of dislocations than for initiating their movement. At the same time, the number of available dislocations appears to decrease with increasing strain.

The different slopes of the log σ vs $1/T$ relation for temperatures above and below the region of about −120°C strongly suggest a change in mechanism of deformation. When yield data for ingot iron[12] are so plotted, all show two linear portions with the change in this same region, and similar slopes among the different heat treatments. In the low temperature regions, the slopes indicate an activation energy of only about 0.15 kcal per mol. The yield of the ingot iron specimen cold drawn 14 pct prior to testing has the same low temperature slope and a lesser slope at warmer temperatures. The yield stress of the National Physical Laboratory 53AF1 iron[3] between room temperature and −73°C, coplotted with the ingot iron specimens, is similar to these and has a slope that is similar.

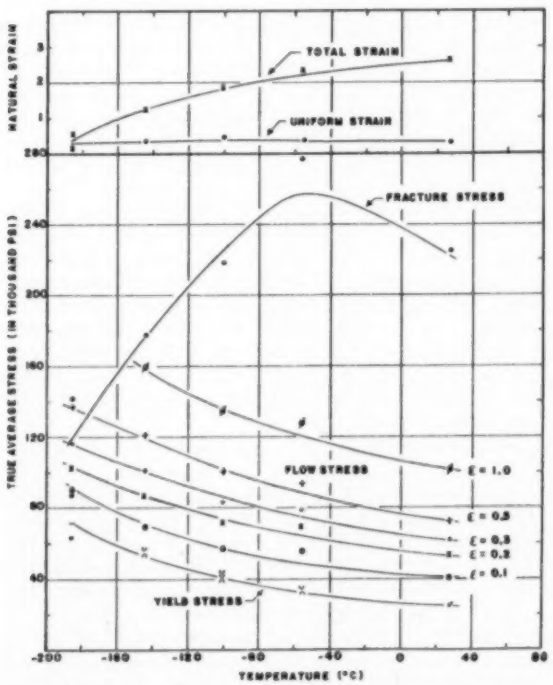

Fig. 4—Effect of temperature upon the yield stress, flow stresses at selected strains, and fracture stress (bottom) and ductility in terms of uniform and total strain (top) of the 2.41 pct Al ferrite with a grain size of ASTM No. 3 to 4 (alloy 28).

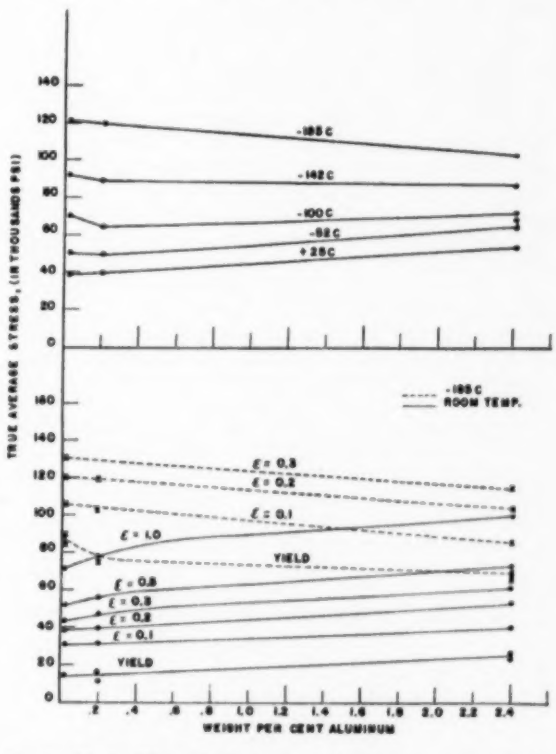

Fig. 5—(Top)—Effect of aluminum on the flow stress at a strain of 0.2 for several temperatures, showing solid-solution softening.

Fig. 6—(Bottom)—Effect of aluminum on the flow stress for several strains at room temperature and at −185°C, showing solid-solution softening.

The change in mechanism could be associated with mechanical twinning at lower temperatures. Among the aluminum ferrites, twins were observed in all alloys deformed at −185°C. Geil and Carwile[12] found twinning up to −120°C, to about the same degree in specimens of ingot iron in all four heat treatments. These twins were present prior to fracture and increased in number as the temperature was decreased. An investigation of ingot iron single crystals by Vogel[9] also showed twinning occurring from −185° up to about −110°C.

Twinning was not observed by Geil and Carwile in the ingot iron cold drawn prior to testing except at fracture edges. However, a change in slope occurred at −150°C in the plot of log σ vs $1/T$ indicating a change in mechanism, and the slope at low temperature is the same as for annealed ingot iron. It is possible that the mechanism, productive of visible twins in the heat-treated ingot iron samples, operated in the deformation of the previously strained material but produced twins of either insufficient length or distinctness to be observed.

As indicated previously, the temperature −100°C marked the beginning of rapid decrease in ductility in all aluminum ferrites. The temperature of decrease in ductility, then, correlates with the temperature at which the mechanism of deformation apparently starts to change.

In the present tests, the role of aluminum in producing solid-solution hardening near room temperature and apparent "solid-solution softening" near liquid air temperature, appears to have two explanations based upon the dual function of aluminum in iron, as follows:

As a solute element, aluminum hardens the iron lattice at room temperature. A different deformation mechanism at low temperatures apparently results in a different effect by aluminum here. If, for instance, the tendency toward twinning is greater for high aluminum contents than for low (in a manner analogous to silicon ferrites[16]), and this tendency manifests itself in a lower activation energy, then, at a sufficiently low temperature, the flow stress for the low aluminum alloys could exceed that for aluminum-rich alloys. In Fig. 7, the slopes of the lines of log σ at $\epsilon = 0.2$ and $\epsilon = 0.5$ vs $1/T$ show a decrease with increasing aluminum content.

The observed decrease in activation energy at low temperatures with increasing aluminum content is also evident at warmer temperatures, Fig. 7. In fact, without a mechanism change between −100° and −150°C, the flow stress for the low aluminum alloys would exceed that for the 2.4 pct Al alloy at a sufficiently low temperature. Higher aluminum contents therefore result in higher k values related, perhaps, to a decreased number of dislocations, but in lower energies necessary to move these dislocations.

The second function of aluminum in iron is that of reducing the soluble oxygen and nitrogen content by forming stable oxides and nitrides. In this role, aluminum may well reduce the energy necessary to move dislocations by reducing solute elements which tend to attach themselves to dislocations or otherwise block their progress. Since the increase in aluminum in these ferrites is accompanied by an increase in the value of k in both temperature regions, it must be presumed that a reduction in the number of dislocations is accomplished.

Table III. Effect of Constant True Strain Rate on Tensile Properties of the 0.02 Pct Aluminum Ferrite at −95°C

	Constant Crosshead Rate, 0.00067 per Sec	Constant Strain Rate, 0.00067 per Sec to Fracture
Yield stress, psi	47,200	47,300
Fracture stress, psi	274,000	165,000
Observed μ, psi	59,000	31,800
Fracture strain	2.77	2.74

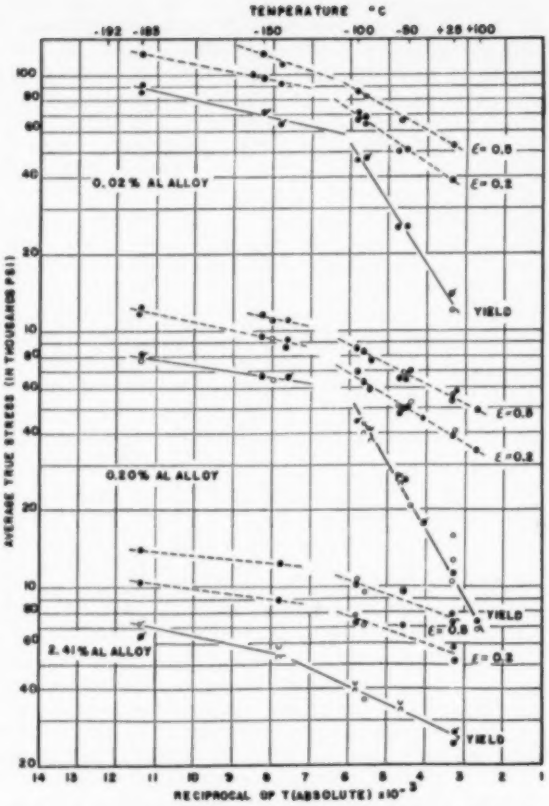

Fig. 7—Yield and flow stresses at strains of 0.2 and 0.5 for the three aluminum ferrites, plotted against reciprocal temperatures, showing a definite change in slope at around −120°C.

Yielding Phenomena and the Effect of Heat Treatment: At room temperature, furnace-cooled specimens of the 0.02 and 0.2 pct Al alloys showed no yield point. The 2.4 pct Al alloy showed some discontinuity at the yield point, while an alloy of 0.2 pct Al with 0.1 pct oxides* suffered a marked drop in load. The degree of the yield-point discontinuity increased with decrease in test temperature.

Quenching following recrystallization rather than furnace cooling had a marked effect upon the yield type. The quenching temperature was generally 700° or 650°C. Quenching not only suppressed the yield point discontinuity at test temperatures down to −29°C, but also markedly lowered yield stresses for the 0.2 pct Al alloy containing oxides. In the other alloys, quenching tended to suppress the yield-effect point which normally appears at lower temperatures but did not noticeably alter stress values.

Some investigation of the effect of cooling rate upon the Rockwell hardness of the 0.2 pct Al alloy with oxides indicated:

* This alloy, No. 25, Table I, picked up oxygen between the hydrogen deoxidation and the vacuum remelt with aluminum. It is not the same alloy as the 0.2 pct Al alloy, No. 26, whose properties are given in Figs. 1 and 3 and which was substantially free of visible oxides.

1—Specimens water quenched after 12 hr at 875°C significantly were harder than those which were furnace cooled.

2—Rapidly cooled specimens softened slightly after reheating at 400°C.

3—This material has been shown not to strain age in 70 min at 425°F following 25 pct cold reduction by rolling.

The association between the higher Rockwell hardness and yield-point depression in quenched specimens of this 0.2 pct Al alloy with oxides is by no means obvious. The production of residual macrostresses upon quenching cannot account for the yield suppression, since specimens, machined from 0.375 to 0.200 in. diameter after quenching, do not differ consistently from specimens requenched after machining although both types of specimens differ decidedly from unquenched specimens.

The lengthy high temperature treatment in dry hydrogen followed by vacuum melting has reduced the carbon and nitrogen contents to below 0.006 and 0.0004 pct, respectively. These appear to be sufficient at low temperatures to cause double yielding. It is hypothesized that contraction of the lattice at lower temperatures results in a greater effectiveness of the residual traces of carbon and nitrogen atoms in locking dislocations. The quench-hardening effect at present must be attributed to a solution effect of AlO or Al_2O_3, since this effect was observable only in the alloy containing 0.1 pct aluminum oxide.

Strain Hardening: The occurrence of a straight-line portion, following maximum load, in the diagrams of true average stress vs natural strain for iron specimens is probably fortuitous because the indicated stress is only an average through the cross section, triaxiality of stress is becoming measureable, and the strain rate is gradually increasing, Table III. When linearity exists, the slope is called the strain-hardening modulus, μ, and is supposed to be a measure of the rate of increase in flow stress with strain or $d\sigma/d\epsilon$. The data of Fig. 1, from maximum load to $\epsilon = 1.0$, show that this slope increases with decrease of temperature. The strain-hardening modulus of each alloy increased slowly from room temperature to about −100°C, after which the increase was more rapid to −185°C.

The apparent higher rate of strain hardening at low temperatures has not been confirmed by penetration-hardness readings at room temperature on specimens strained at low temperatures. A series of specimens, tested at temperatures from room temperature to −185°C, were sectioned longitudinally and microhardness measurements taken at comparable values of ϵ. The microhardness was found to be lower in specimens strained at low temperatures rather than higher, Fig. 8. Furthermore, the strain at maximum load, Fig. 1, decreased appreciably with decrease in test temperature. This is further evidence of lesser strain hardening at low temperatures,[13] since it can be shown that the strain at maximum load is numerically equal to the strain hardening exponent n in the equation $\sigma = k\epsilon^n$.

Strain Rate: It was stated previously that a constant rate of total crosshead movement, or of elongation of the specimen during testing, results in a progressive increase in the true strain rate. This increase is particularly great during necking and it will be shown that this appreciably increases the flow stress at strains of 1.0 and greater.

A mechanical device was constructed which allowed the rate of straining to be adjusted to a specific true strain rate at any point during the test. An indicating needle, driven by the autographic recorder, revolved once for every 0.010 in. of decrease in diameter. Mounted behind and coaxially with the needle was a pacer disk. The rate of rotation of the disk was decreased during test so that its rate, the true strain rate of 0.00067 per sec., was dependent upon the existing diameter of the specimen. Explicitly, the conditions for constant true strain rate $\dot{\epsilon}$, are

$$\epsilon = 2\ln\frac{d_o}{d_i}, \frac{d\epsilon}{dt} = -2\frac{\frac{d(d_i)}{dt}}{d_i} = 0.00067 \text{ per sec}$$

or

$$\frac{d(d_i)}{dt} = -\frac{0.00067}{2}d_i$$

where d_i is the instantaneous diameter and d_o is the original diameter.

The number of available specimens prohibited testing at both a constant crosshead rate and a constant true strain rate. It was found that during a test, conducted at a constant rate of crosshead movement (0.00067 in. per sec), the rate could be decreased until the true strain rate was obtained, held for several thousandths decrease in diameter, and then returned to the original rate without impairing the test. This procedure was followed during a number of tests, but decreases to the true strain rate were limited, in most tests, to one or two so that this variation in strain history would not be significant.

Since specimens were tested in duplicate or triplicate at several test temperatures, it was hoped that the values of $\sigma/\sigma_{\dot{\epsilon}}$** could be obtained at enough strains to allow an approximate σ vs ϵ diagram for $\dot{\epsilon} = 0.00067$ per sec to be constructed. This information at several temperatures would allow some evaluation to be made. Actually the data were not sufficient for complete analytic treatment although a few conclusions can be reached.

** $\sigma/\sigma_{\dot{\epsilon}}$ is the ratio of the stress for a particular strain, reached under conditions of constant rate of elongation, to the stress for the same strain reached under conditions of constant strain rate.

The data show the marked effect of the increasing true strain rate, produced by a constant rate of crosshead movement, upon the fracture stress and apparent strain hardening. It is interesting to note that the strain at fracture is not greatly affected, indicating that if fracture occurs at a limiting stress

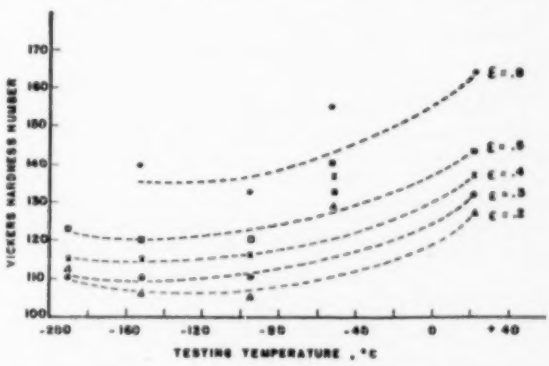

Fig. 8—Microhardness test data (Vickers pyramid in Tukon tester) for the 0.02 pct Al ferrite which was strained different amounts at five different temperatures. Despite scatter of these plotted averages and apparently high values for the −52°C specimens, the data show lower strain hardening for lower temperatures of straining.

value, the limiting value also must be dependent upon the rate of deformation.

The same tests gave the information presented in Table IV, showing the change in the true strain rate, $\dot{\epsilon}$, produced at different strains by a constant rate of crosshead movement of 0.00067 in. per sec and the resulting increase in flow stress, $\sigma/\sigma_{\dot{\epsilon}}$. These are average values from several tests. The effect of testing at a constant rate of crosshead motion, upon both the true strain rate ($\dot{\epsilon}$) at large strains and the measured flow stress, appears to be appreciable. It must be emphasized that the effect of strain rate was by no means evaluated completely in this investigation. The results obtained, however, clearly indicate that strain rate is an important variable in these data and its explicit control or evaluation must be made if the fundamental parameters of deformation are to be measured.

Aluminum-Silicon Ferrites: Early in this investigation, four alloys were produced by a preliminary aluminum deoxidation of electrolytic iron rather than by hydrogen deoxidation. The air melt crucibles contained some silica and, as a result, these first four alloys were essentially aluminum-silicon ferrites containing up to 0.2 pct Si. These should be regarded as less pure than the aluminum ferrites because they did not receive the extended dry-hydrogen treatments at 1150°C to remove oxygen, carbon, nitrogen, and sulphur to which the material for the binary aluminum ferrites was subjected. Nevertheless, their properties put these alloys in the same category as the purest alloys.

The chemical analyses for these alloys are listed in Table I. In Fig. 9, yield and flow-stress values at different temperatures for both aluminum and aluminum-silicon ferrites are plotted against the sum of the percent atomic solid-solubility limits of the solutes. Fig. 9 indicates that, in the absence of oxygen, the effect of silicon in the usual steelmaking range of compositions is only that of another solute. The apparent "solid-solution softening" observed for the aluminum ferrites is not altered by the presence of these small amounts of silicon.

The most significant finding from the tests of these alloys is that silicon, as such, is not embrittling, at least in small amounts. One specimen of an alloy with 0.27 pct Al and 0.09 pct Si had a total strain of 0.8 (55 pct reduction of area) at −185°C.

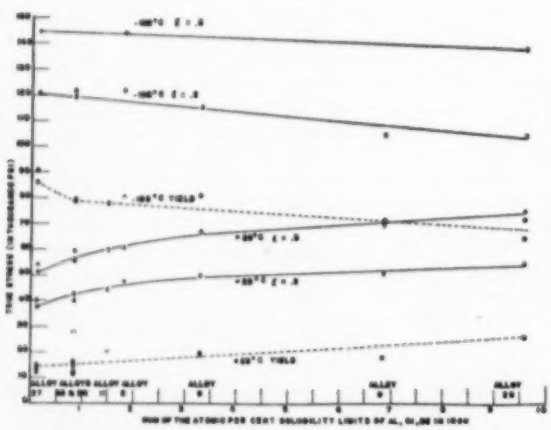

Fig. 9—Yield and flow stresses at room temperature and at −185°C for both aluminum and Al-Si ferrites plotted against the sum of the fraction of solubility of these solutes.

Table IV. Increase in True Strain Rate, $\dot{\epsilon}$, and in Flow Stress, $\sigma/\sigma_{\dot{\epsilon}}$, Produced by Constant Rate of Crosshead Movement; 0.02 Pct Aluminum Ferrite at −95°C

ϵ	$\dot{\epsilon}$	$\frac{\dot{\epsilon}}{0.00067}$	$\sigma/\sigma_{\dot{\epsilon}}$
0	0.00067	1	1
0.4	0.0026	3.9	1.02
0.9	0.01	15	1.09
1.6	0.035	52.5	1.16
2.0	0.059	88.5	1.25
2.2	0.074	112	1.32
2.3	0.086	129	1.4

Conclusions

1—Alloys of vacuum-melted hydrogen deoxidized iron with from 0.02 to 2.41 pct Al have appreciable ductility in uniaxial tension at −185°C. However ductility is rapidly decreasing at this liquid air temperature and complete brittleness at some lower temperature may be assumed. Since the amount of aluminum in these oxygen and nitrogen-free alloys was not of appreciable effect, it is concluded that the beneficial effect of aluminum in semikilled steel may be attributed to its deoxidizing and grain-size effects.

2—Yield stress and flow stress of these alloys at fixed natural strains increased with decrease of test temperature. The more rapid rate of increase of these stress values for low aluminum-content ferrites caused apparent "solid-solution softening" below about −150°C, i.e., increased aluminum content reduced the flow stress at a given strain. At room temperatures, the usual solid-solution hardening by aluminum was found.

3—Fracture stress and energy to fracture increased from +100° to −100°C because of the increase in flow stress and constancy of total deformation. Below −100°C, fracture stress and energy to fracture decreased rapidly as ductility and related strain hardening diminished. The rate of decrease of fracture stress was less for the higher aluminum ferrites in which the rate of loss of ductility was lower.

4—As much as 0.2 pct Si in aluminum ferrites acts only as another solute element. It does not specifically embrittle iron under conditions of uniaxial static tensile deformation.

5—There appears to be a change in mechanism of deformation in ferrites at about −100°C as indicated by an appreciable decrease in apparent activation energy for deformation below this temperature. The effect is similar in ferrites of all compositions studied. The temperature range for this change is the same as that in which ductility starts to decrease and mechanical twinning becomes apparent.

6—Low aluminum ferrites show no yield point at room temperature but develop yield-point discontinuities at lower temperatures. The yield-point discontinuity is reduced by quenching from 650°C even though these were pure alloys and micrographically were single-phase structures.

7—The constant rate of crosshead motion used in most of the tests caused greatly increased flow stresses at large strains as compared to the flow stress for constant true strain rate. The slopes of the stress-strain line and the fracture-stress line were also greatly affected but total strain or ductility was not.

Acknowledgment

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Technical Note

Locations and Sizes of Interstitial Holes in the Alpha-Uranium Lattice

by A. J. Opinsky

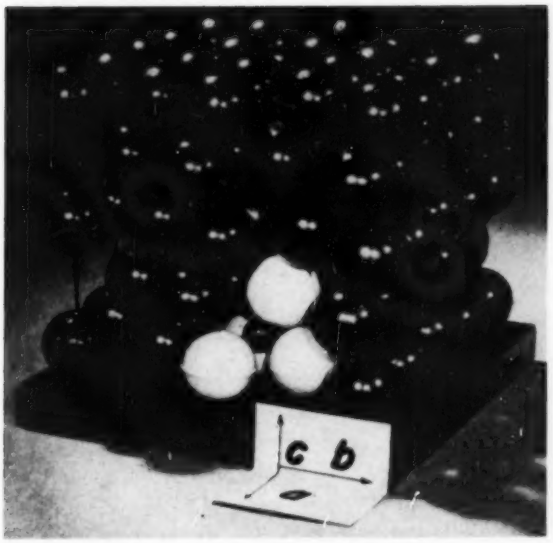

Fig. 1—Model of uranium lattice illustrating interstitial surrounded by four atoms.

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THE purpose of this note is to indicate the locations and sizes of various interstitial holes in the orthorhombic uranium lattice based on a hard-sphere model. First, plane configurations of three and four atoms will be examined, and then the true interstices surrounded by four atoms in space and by five atoms in space will be obtained. The data and notation contained in a report by Tucker[1] will be used.

Plane Configurations

Fig. 1 presents the orthorhombic uranium lattice made of spheres touching at the closest interatomic distance. The three light-colored spheres in the *ab* plane are illustrative of the first type of configuration to be examined. If one edge of the unit cell is taken as the origin, the three atoms can be represented by the vectors $[0i + 0j + 0k]$, $[ai + 0j + 0k]$ and $[(a/2)i + (b/2)j + 0k]$ where i, j, and k are respectively parallel to a, b, and c. In the future, this notation will be shortened to [000], [a00] and [a/2 b/2 0], respectively. A vector $R = [r\ s\ t]$ describes the position of the hole surrounded by the atoms. Since the center of the hole must be equidistant from the three atoms

$$|R|^2 = r^2 + s^2 + t^2 = (r-a)^2 + s^2 + t^2 = (r-a/2)^2 + (s-b/2)^2 + t^2.$$

$R = [a/2\ (b/4) - (a^2/4b)\ 0]$ is the solution of these

Table I. Location of Various Interstitial Holes in the Uranium Lattice (The Origin is at the Edge of the Unit Cell)

Type	Atoms Associated	Hole Location: General Terms	Hole Location: Numerical	Diameter of Hole, A
3 atoms in *ab* plane	[0 0 0] [*a* 0 0] [*a*/2 *b*/2 0]	[*a*/2 *b*/4 − (*a*²/4*b*) 0]	[1.43 1.12 0]	0.88
3 atoms not in *ab* plane	[0 0 0] [*a*/2 *b*/2 0] [*a*/2 *b*/2-2*by* *c*/2]	Solved numerically	[0.44 1.60 0.88]	1.00
4 atoms in corrugated plane	[0 0 0] [*a* 0 0] [0 −2*by* *c*/2] [*a* −2*by* *c*/2]	[*a*/2 − *by* *c*/4]	[1.43 −0.62 1.24]	1.22
4 atoms in space	[0 0 0] [*a*/2 *b*/2-2*by* −*c*/2] [−*a*/2 *b*/2-2*by* −*c*/2] [0 −2*by* −*c*/2]	[0 (*a*²/4*b*) + (*b*/4) − 2*by* −(*c*/4 + (*by*/*c*) (*a*²/*b* + *b* − 4*by*))]	[0 0.58 −1.84]	1.10
5 atoms in space	[0 0 0] [*a* 0 0] [0 −2*by* *c*/2] [*a* −2*by* *c*/2] [*a*/2 −*b*/2 0]	[*a*/2 (*a*²/4*b*) − (*b*/4) (*c*/4 − (*by*/*c*) (*b* − *a*²/*b* − 4*by*))]	[1.43 −1.12 0.99]	1.36

equations, or, evaluated, [1.43 1.12 0]. The distance from [0 0 0] to [1.43 1.12 0] is 1.82Å; the diameter of the hole, using 1.38Å as the atomic radius of uranium, is thus 0.88Å. The data for this and other plane configurations similarly calculated are listed in Table I. For these plane configurations, only one location is calculated and no attempt will be made to place them in the unit cell.

Interstitial Surrounded by Four Atoms in Space

Fig. 1 also illustrates the interstitial amid four atoms in space as the dark marble surrounded by the four light-colored spheres. The results of a sample calculation are given in Table I. For counting purposes, this interstitial will be considered as lying between a base of three atoms in the *ab* plane and the near-neighbor apex atom above (or below). There are two tetrahedral configurations for each apex atom (one pointing in the general direction of $+c$ and the other, $-c$). There are really four orientations of these tetrahedral configurations, since whenever a jump of $c/2$ is made, the sign of the j component when the apex is at [0 0 0] must be reversed. This can be seen by inspection of Fig. 1.

Fig. 2—Model of uranium lattice illustrating interstitial surrounded by five atoms.

Table II. Locations of Various Interstitial Holes in the Uranium Unit Cell

Surrounded by Four Atoms in Space*	Surrounded by Five Atoms in Space**
[0 *u* ½ + *v*]	[0 ½ − *U* ½ − *V*]
[0 *u* −*v*]	[0 ½ − *U* *V*]
[0 −*u* *v*]	[0 ½ + *U* −*V*]
[0 −*u* ½ − *v*]	[0 ½ + *U* ½ + *V*]
[½ ½ + *u* ½ + *v*]	[½ −*U* ½ − *V*]
[½ ½ + *u* −*v*]	[½ −*U* *V*]
[½ ½ − *u* *v*]	[½ *U* −*V*]
[½ ½ − *u* ½ − *v*]	[½ *U* ½ + *V*]

* Where $u = (a^2/4b^2) + 1/4 - y$; $v = (by/c^2)\ (a^2/b + b - 4by)$.
** Where $U = \frac{1}{4} - (a^2/4b^2) - y$; $V = (by/c^2)\ (b - a^2/b - 4by)$.

Since there are two interstitials per atom and four atoms per unit cell, there are eight four-atom interstitials in the unit cell. Table II locates these interstitials in the unit cell given by Tucker.[1]

Interstitial Surrounded by Five Atoms in Space

Fig. 2 illustrates the five-atom interstitial as the dark marble amid the five light-colored spheres. These atoms form a slightly skewed pyramid, as is shown by the following calculation: Consider the four atoms lying in the corrugated plane; their center of gravity, Table I, is $[a/2 - by\ c/4]$. Since the apex atom will be at $[a/2\ b/2 - 2by\ c/2]$, the vector V joining the apex and the center of gravity of the base will be $V = [0\ (b/2) - by\ c/4]$. On the other hand, the normal to corrugated plane is given by the cross-product of two vectors in the plane, such as $[0 - 2by\ c/2] \times [a\ 0\ 0]$ or $N = [0\ ac/2\ 2aby]$. If the pyramid is regular, N and V will be parallel, or $N \times V = 0$. Thus, $N \times V = [(ac^2/8 - ab^2y + 2ab^2y^2)\ 0\ 0]$; $N \times V = 0$ only if $y = 0.116$. But for uranium, $y = 0.105 \pm 0.005$;[1] thus the pyramid is slightly skewed, with the angle between N and V being approximately 1.5°.

Table I lists the results of the calculation of one five-atom interstitial. There are again two interstitials associated with each apex atom, or eight interstitial sites per unit cell, Table II. Since the pyramid is skewed, there are four non-parallel orientations of the pyramidal configuration. This is the largest interstice, having a diameter of 1.36Å.

[1] C. W. Tucker, Jr.: *The Crystal Structures of Metallic Uranium.* AECD—2716.

Columbium-Vanadium Alloy System

by H. A. Wilhelm, O. N. Carlson, and J. M. Dickinson

On the basis of microscopic studies, melting-point observations, and X-ray analyses, a phase diagram is proposed for the Cb-V system. A complete series of solid solutions is formed with a minimum in the solidus at 1810°C near 35 wt pct Cb. No compounds or intermediate phases were found in the system above 650°C.

THERE is an ever increasing need for better structural metals and alloys for use in nuclear reactors. In addition to the normal properties of engineering structural materials, such as high temperature strength, resistance to corrosion, and fabricability, the nuclear properties of the material must be considered. In a nuclear reactor it is important to conserve neutrons, so a material which removes these neutrons from the reaction excessively is considered to have unfavorable nuclear properties. In nuclear-reactor design the engineer must have nuclear as well as other data available on alloys in order to make a wise selection of materials. Due to the fact that many of the common structural materials have undesirable nuclear properties, it is vital that new alloys of metals having more favorable nuclear properties be investigated.

Columbium and vanadium are both high melting metals, both exhibit resistance to chemical attack, and no great difficulty is encountered in fabricating them into desired shapes. With proper treatment both metals can be cold rolled extensively without failure. In addition they have desirable nuclear properties for certain types of reactors. Therefore, the alloys of columbium and vanadium should be of interest in the atomic energy program. Since an alloy-development program is enhanced by a knowledge of the phase equilibria of the components, this investigation was undertaken to establish the phase diagram for the Cb-V system.

According to the Hume-Rothery rules for alloying,[1] the chemical similarity, crystal structure, and atomic-size factor are favorable for a complete series of solid solutions for this system. Both elements are in the same family of group V of the periodic table and thus are quite similar chemically. The crystal structures of columbium and vanadium are compatible for extensive solid solubility, since both have body-centered-cubic structures. The atomic diameters of columbium and vanadium are 2.85 and 2.62A, respectively. This difference of slightly more than 8 pct is well within the 15 pct maximum difference allowed for extensive solid solubility.

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Contribution No. 304 from the Institute for Atomic Research and Department of Chemistry, Iowa State College, Ames, Iowa. Work was performed in the Ames Laboratory of the Atomic Energy Commission.

Experimental Procedures

Source of Materials: Columbium powder and sheet trimmings were obtained from the Fansteel Metallurgical Corp. According to the manufacturer the metal contains less than 1 pct impurity. An analysis of the metal showed approximately 1800 ppm C in the powder while the sheet trimmings contained less than 500 ppm C. Spectrographic analysis showed minor amounts of Ca, Cr, Fe, Mn, Si, Ti, V, and Zr in both forms of the columbium.

No commercial source of vanadium having the ductility and purity desired was available to the authors at the beginning of this investigation. As a result, all of the vanadium used in this study was prepared by the bomb reduction of vanadium pentoxide with calcium employing the method reported by Long.[2] Yields of massive vanadium normally were about 80 pct. Chemical analysis of the vanadium prepared in this manner showed the presence of 200 to 500 ppm N and 800 to 1000 ppm C. Minor amounts of Ca, Fe, Mn, Si, Zr, Cr, and Cb were detected by spectrographic analysis. This vanadium metal was ductile and was cold rolled into 5 mil sheet. Annealing was not necessary during this rolling and the metal retained its cold-rolling characteristic after arc-melting.

Preparation of Alloys: The Cb-V alloys were prepared by melting pieces of vanadium sheet together with columbium in the form of sheet or pellets of powder. The melting was carried out under argon in conventional arc-melting equipment employing a tungsten electrode and a water-cooled copper crucible. Each alloy was remelted three or four times, inverting the alloy after each melting in order to assure complete mixing. Alloys normally were obtained as round flat disks, weighed approximately 70 grams, and had roughly the shape of a disk 1½ in. in diameter and ¼ in. thick. Half of each alloy

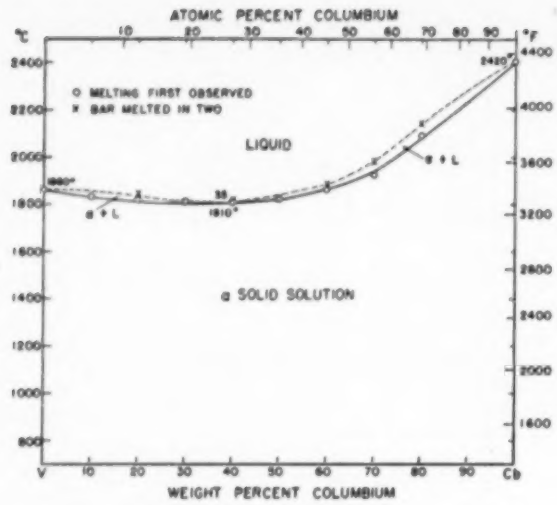

Fig. 1—Cb-V phase diagram.

was given a homogenization treatment in a graphite crucible at approximately 1600°C for 4 hr to eliminate possible solid-solution coring. Since some of these alloys reacted with the graphite during this treatment, the remaining portion of the alloys was annealed at a lower temperature, 1075°C, for a longer time, 48 hr, to condition them for further investigation and study.

By accurately weighing the component materials before melting and the resulting alloy afterwards, a small loss in weight was usually observed. If this loss were attributed entirely either to the columbium or the vanadium, the shift from the intended composition would never have exceeded 0.3 pct. Thus the compositions have been assigned on the basis of that calculated from the charge. No chemical analyses were performed on the final alloys.

Metallography: Alloys of the Cb-V system were ground and polished in wet operations. The rough grinding was performed on a belt sander with a stream of kerosene flowing over the sample. The final grinding operations were performed on a sander in the presence of water. The alloys were polished on either microcloth or billiard cloth using Linde *A* polishing compound. The alloys were quite resistant to most etchants but it was found that by immersion in a mixture of 1 part H_2SO_4, 1 part 48 pct H_2F_2, 2 parts HNO_3, and 1 part H_2O most of the structures could be revealed. The microstructures were examined and photographed with ordinary illumination employing a Bausch and Lomb research metallograph.

X-Ray Methods: Filings were taken from the alloys annealed at 1075°C. These were further annealed for 113 hr at 650°C in sealed evacuated quartz capillaries. X-ray diffraction patterns were obtained with a 114.7 mm diameter Debye-Scherrer camera. The resolutions of the lines in the back-reflection region were generally very poor and consequently it was not feasible to use a precision back-reflection-type camera. Accurate lattice constants were obtained by using both reflection regions of the Debye-Scherrer camera and by extrapolating the lattice constant determined for each line against the Nelson-Riley[3] function

$$\tfrac{1}{2}\left[\frac{\cos^2\theta}{\sin\theta}+\frac{\cos^2\theta}{\theta}\right].$$

The film was corrected for both linear and nonlinear film shrinkage using a calibration method[4] involving a device which punches fiducial marks on the unexposed film. After processing the film, the positions of the marks were measured and corrections made for changes in film dimension.

Melting Range Temperatures: The solidus for the Cb-V alloy system was determined by a modification of the method of Pirani and Alterthum.[5] Briefly, this method consists of heating a bar of the alloy by passing high electric current through it while observing the melting temperature by means of an optical pyrometer focused on a small hole that had been drilled in the bar. In some cases the hole did not fill immediately with metal when melting first started. In those instances the bar was heated until it melted in two. The point at which liquid was first observed was taken as the solidus temperature and the point at which the bar melted in two was taken as an indication of the liquidus temperature. A correction was made for absorption by the sight glass using the relationship[6]

$$\frac{1}{T}-\frac{1}{T_a}=\log\frac{A}{c_2}\log\lambda$$

where T is the true absolute temperature, T_a is the apparent or observed absolute temperature, c_2 is a

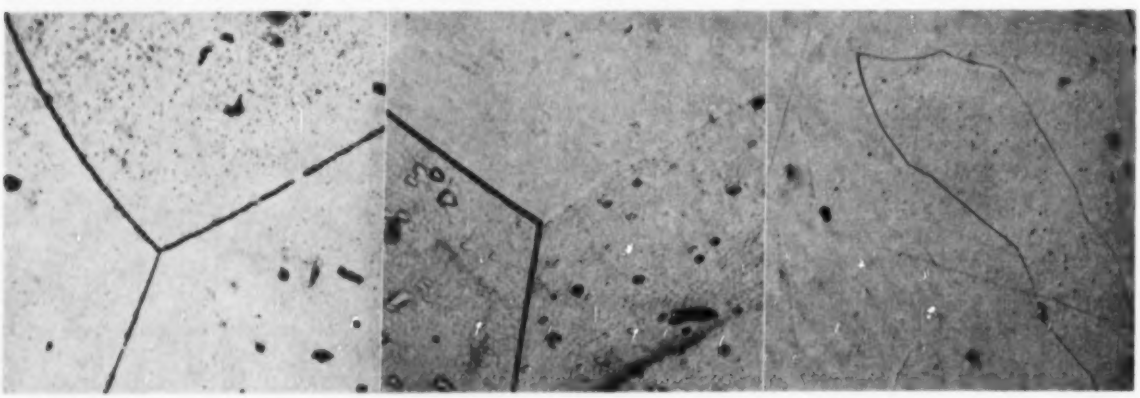

Fig. 2—Vanadium metal arc-melted and annealed for 48 hr at 1075°C. Etchant, 2 parts HNO_3, 1 part H_2SO_4, 1 part H_2F_2 and 1 part H_2O. X250. Area reduced approximately 20 pct for reproduction.

Fig. 3—Columbium sheet as-arc-melted. Etching pits within grains are a result of heavy etching. Etchant, aqua regia plus 5 pct H_2F_2. X250. Area reduced approximately 20 pct for reproduction.

Fig. 4—50 pct Cb alloy as-arc-melted. Etchant, 2 parts HNO_3, 1 part H_2SO_4, and 1 part H_2F_2. X250. Area reduced approximately 20 pct for reproduction.

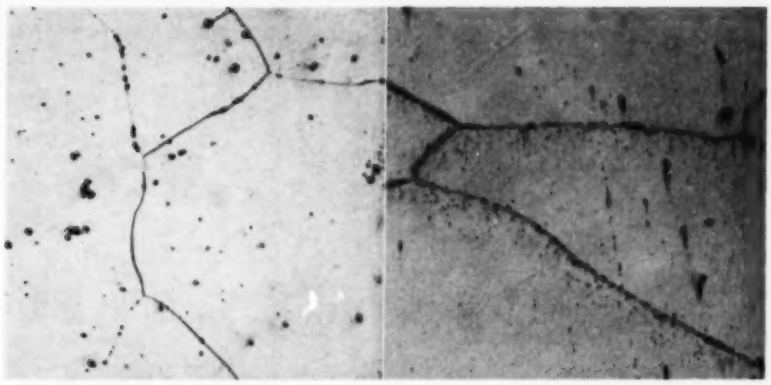

Fig. 5—30 pct Cb alloy annealed at 1075°C for 48 hr. One-phase structure. Etchant, 2 parts HNO_3, 1 part H_2SO_4, and 1 part H_2F_2. X250. Area reduced approximately 20 pct for reproduction.

Fig. 6—30 pct Cb alloy annealed at 900°C for 170 hr. One-phase structure. Etchant, 2 parts HNO_3, 1 part H_2SO_4, and 1 part H_2F_2. X250. Area reduced approximately 20 pct for reproduction.

constant 14,320, A is the percent transmission of sight glass, and λ is 0.65 microns.

Experimental Results

The phase diagram shown in Fig. 1 has been constructed from data obtained by X-ray diffraction studies, metallographic examination, and observations on melting temperatures.

Metallographic Observations: All of the alloys in the solid region of the Cb-V system were found to be one phase, both in the as-arc-melted condition and after various annealing treatments. Fig. 2 is a micrograph of the unalloyed vanadium metal used in these studies and shows the impurities characteristically present in this vanadium. The columbium sheet was microscopically quite clean as can be seen in Fig. 3 . The etching pits and heavily outlined grain boundaries are the result of heavy etching and are characteristic of columbium and many of the Cb-V alloys. Unannealed alloys at 10 pct intervals across the systems were examined and all were found to have single-phase microstructures, similar to that shown for the 50 wt pct alloy in Fig. 4. Since the arc-melting process is essentially a high temperature quench, this may not be representative of the structure at lower temperatures; consequently, the entire series of alloys was annealed for 48 hr at 1075°C, furnace cooled, and examined microscopically. All of the alloys were again found to be one phase, resembling the 30 pct Cb alloy shown in Fig. 5.

Realizing that the temperature selected for the above annealing treatment may have been too high to obtain evidence of a true second phase, the 30, 50, 60, and 70 pct Cb alloys were subsequently annealed for 170 hr at 900°C, furnace cooled, and examined microscopically. No change in the one-phase appearance of any of the alloys was noted. Fig. 6 is a micrograph of the representative 30 pct Cb alloy from this series. Rostoker and Yamamoto[7] report microscopic evidence for a possible σ phase in a V-40 pct Cb alloy which had been annealed for 170 hr. However, no microscopic evidence for such a second phase could be detected in any of the alloys which were annealed at 900°C in the present investigation.

X-Ray Data: As described earlier, the lattice constants of the Cb-V alloys were determined from powder patterns. The massive alloys were annealed at 1075°C for 48 hr and filings taken from these were given an additional anneal at 650°C for 113 hr. Fig. 7 is a plot of atomic percent columbium against the measured lattice constants of the alloys. This curve exhibits a very slight positive deviation from Vegard's law but has no discontinuities. However this deviation might not be real, since, it must be remembered, the composition data employed in this graph were assumed to be that of the charge placed in the melting crucible.

In a solid-solution system of this type, the possibility of order-disorder or of the aforementioned σ phase formation should be considered. From the X-ray data no evidence was found for the existence

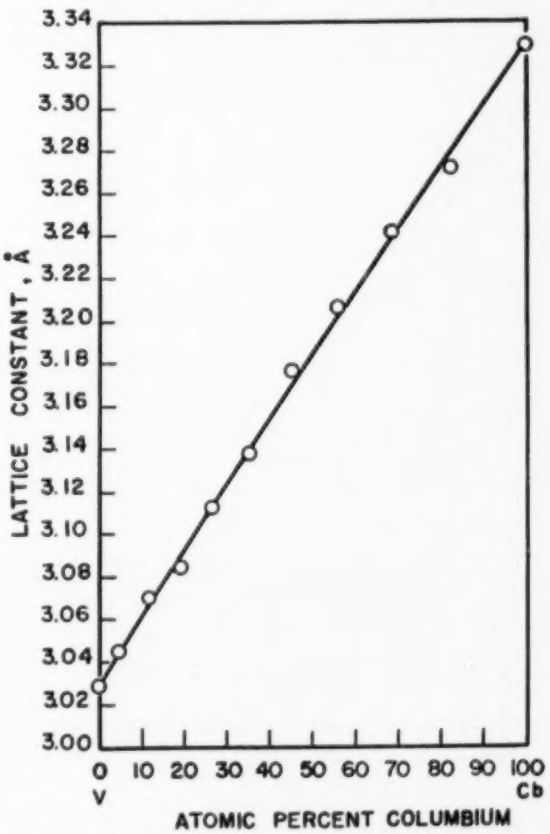

Fig. 7—Lattice constant vs atomic percent columbium.

of either. The powder diffraction patterns taken on all the alloys in the series produced no evidence for superlattice lines which might have indicated ordering, nor were there any lines present signifying the presence of a σ phase. Thus, on the basis of the above metallographic and X-ray evidence it was concluded that columbium and vanadium form a complete series of solid solutions at temperatures down to 650°C.

Thermal Data: The liquidus and solidus points as determined by the melting-bar method are shown in Table I and have been plotted on the phase diagram, Fig. 1. The solidus has a long, almost flat section extending from about 20 to 50 pct Cb and in this region the liquidus is almost identical with the solidus. The reproducibility of the melting temperature was very good, in most cases within ±15°C. The minimum melting temperature obtained was 1810°C, the composition at which this minimum occurs is in the neighborhood of 35 pct Cb.

Since vanadium has an appreciable vapor pressure near its melting point, a film of vanadium metal fogged the sight glass during the liquidus determinations. A temperature correction was made for this film by measuring the transmission of the sight glass after each experiment and applying the formula given under experimental procedures. In those instances where the transmissions were found to be less than 80 pct, the corrections were considered excessive and the data obtained were not used in constructing the phase diagram.

Since microstructures and X-ray data supplied strong evidence for a continuous solid solution across this system, there was little reason to make a detailed cooling-curve investigation. However, a cooling curve was run on the 30 pct Cb alloy and no thermal arrests were found between 1100°C and room temperature.

Physical Properties

The physical properties of the alloys are greatly influenced by the quality of the metals used. Alloys prepared from the columbium powder which contained 1800 ppm C were very brittle and difficult to machine or work. On the other hand alloys made from the columbium sheet containing 500 ppm C were ductile and easily machined. Alloys containing 5, 10, and 40 pct Cb were successfully cold rolled into 10 mil sheet without serious edge cracking and with no intermittent annealing.

Hardness values were measured on the alloys both as-arc-melted and after annealing and have been plotted against weight percent columbium as shown in Fig. 8. In all cases, it will be noted, the curves have a flat maximum and alloys made with

Table I. Melting Temperatures of Cb-V Alloys

Composition, Wt Pct Cb	Temperature At Which Liquid Was First Observed, °C	Temperature At Which Bar Melted in Two, °C
0	1858, 1861*	
10	1838	
20	1813, 1819, 1821	1840
30	1811, 1820	
40	1807	1812
50	1821, 1824	
60	1859, 1864	1874
70	1928, 1929	1980
80	2080	2140
100	2420	

* Duplicate and triplicate melting-point determinations were made on some of the samples.

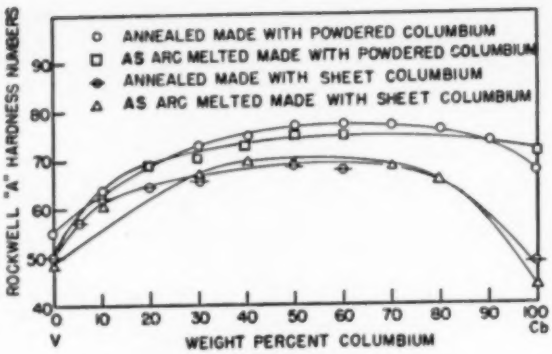

Fig. 8—Hardness vs composition for Cb-V system.

columbium are considerably harder than those made from the columbium sheet.

Corrosion tests were made on the 5, 10, and 50 pct Cb alloys, columbium sheet, and vanadium sheet in a stainless-steel sealed vessel. After 375 hr in 178°C distilled water there was very little change in the appearance of the alloys and the weight changes were all within experimental error, 0.0003 grams.

Similar tests were then carried out at 250°C for 60 hr. Except for the vanadium specimen which was covered with a black film, the specimens were only slightly tarnished. However, weight changes were small on all of the specimens tested. A further test at 330°C for 60 hr produced a black film on all specimens but none had disintegrated. The vanadium and the 5 pct Cb alloys showed the most attack.

Summary

Columbium and vanadium have been shown, by means of metallographic examination and X-ray studies, to form a complete series of solid solutions. No evidence of a second solid phase was found for this system at temperatures above 650°C. The solidus curve has a long, nearly flat minimum extending from about 10 to 50 pct Cb with the minimum temperature at about 1810°C near the composition of 35 wt pct Cb. The liquidus curve lies very close to the solidus curve throughout the system examined.

Alloys prepared from columbium powder were brittle and could not be cold rolled, whereas those prepared from columbium sheet were much softer and more ductile. This difference is believed to be due to impurities in the columbium powder.

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Some Observations on Grain Boundary Shearing During Creep

by Bernard Fazan, Oleg D. Sherby, and John E. Dorn

McLean's technique was employed to determine the effect of temperature on the contribution of grain boundary shearing to the total creep strain in pure aluminum over the range of 610° to 747°K. The contribution of grain boundary shearing at the same stress was found to depend upon a temperature-compensated time, $te^{-\Delta H/RT}$, where the activation energy ΔH was practically identical with that for total high temperature creep.

SEVERAL outstanding investigations by McLean[1-4] have demonstrated that the total creep strain in polycrystalline aluminum at 473°K arises from microscopically detectable slip, subgrain tilting, and grain boundary shearing. Whereas microscopically detectable slip arises from dislocations that move out of the grains, subgrain tilting is due to entrapment of dislocations of like sign in subgrain boundaries. Consequently, creep in polycrystalline aggregates appears to be the resultant of two mechanisms, namely, migration of dislocations and grain boundary shearing. This suggests that an appropriate law for creep should contain two additive terms

$$\epsilon_t = \epsilon_d + \epsilon_{gb} \qquad [1]$$

where the total creep strain, ϵ_t, depends on the sums of the creep strains arising from motion of dislocations, ϵ_d, and from grain boundary shearing, ϵ_{gb}.

But extensive investigations[5] over wide ranges of alloys, grain sizes, temperatures, and stresses have shown that the total creep strain is quite accurately dependent on the single term functional relationship

$$\epsilon_t = f(\theta), \sigma = \text{constant} \qquad [2]$$

where σ is the stress (constant),* $\theta = te^{-\Delta H/RT}$, t is the time under stress, ΔH is the activation energy, R is gas constant, and T is absolute temperature.

Since Eq. 2 was found to be valid over those ranges where appreciable grain boundary shearing is observed, it appears probable that the activation energy for grain boundary shearing might be identical with the activation energy for migration of dislocations. Thus, under a given stress, it could be thought that

$$\epsilon_t = f_d(\theta) + f_{gb}(\theta) = f(\theta)$$

where f_d and f_{gb} represent the appropriate functions for each mechanism of creep. The primary objective of this investigation was to obtain a direct check on the accuracy of this hypothesis.

Experimental Procedure and Results

Rolled aluminum sheet (99.987 pct Al), 0.100 in. thick, was used in this investigation. Creep specimens having a 0.25 in. wide, 3 in. long gage section were so machined that their axes were in the rolling direction. All specimens were annealed for 9 hr at 894°K before testing to produce a mean grain diameter of about 0.1 in. One face of each specimen was polished electrolytically in 10 pct HBF_4. In order to permit accurate measurements of grain boundary shearing, the polished surface was lightly scribed with a ruling machine accurate to ±0.0001 in. to give a uniform grid of lines which were 0.01 in. apart. Although it was thought that surface working might modify the creep behavior in the vicinity of the scribed grid lines, this technique nonetheless was employed because such local differences in creep properties should not modify seriously the fractions of the total creep strain due to grain boundary shearing or to migration of dislocations. All creep tests were conducted under constant load with an initial stress of 250 psi. The specimen tem-

* Eq. 2 is valid for either constant stress or constant load tests; the total creep curve, however, is different (i.e., the function, f, is different) for each type of test.

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Table I. Conditions of Test. Stress, 250 Psi

Number	Temperature, °K	Total Creep Strain, ϵ_t
1	610	0.037
2	747	0.037
3	610	0.070
4	747	0.070
5	610	0.110
6	640	0.110
7	700	0.110
8	747	0.110

perature, as measured by two calibrated thermocouples attached at the two ends of the gage section, was held to within ±2°K of the reported temperature. The total creep strain during the course of a test was measured by means of a simple rack and pinion extensometer sensitive to 0.01 pct for a 2 in. gage length. After several exploratory tests, eight tests were made, see Table I.

The tests at various temperatures from 610° to 747°K were interrupted at prescribed total strains, ϵ_t, of 0.037, 0.070, and 0.110, and the specimens were immediately removed from the creep machine for metallographic analyses and measurements of grain boundary shearing.

The technique employed for determining the amount of grain boundary shearing was that introduced by McLean.[1-4] An example of grain boundary shearing is shown in Fig. 1. The vertical displacement of a horizontal grid line at a grain boundary is designated by k. The greatest shear displacements were found on those grain boundaries that ran about 45° to the specimen axis. But wide ranges of k values were obtained for different boundaries of the same orientation (relative to the specimen axis) suggesting that grain orientations also affect grain boundary shearing. Furthermore, in many of the cases examined, the values of k varied appreciably even over a single grain boundary.

As shown in Fig. 2 at regions where grain boundary migration took place, the grid lines appear to have been bent. It was assumed that the appearance of bending was due to alternate grain boundary migration and shearing. An example of this on a coarse scale is shown in Fig. 3. Consequently, for such cases of bent grid lines, k was determined by measuring the vertical displacement of the grid line over the entire region where grain boundary migration took place. An extreme example of alternate grain boundary shearing and migration on a very fine scale is illustrated in Fig. 4. It was also observed that polygonization occurred preferentially along the grain boundaries rather than in the grain cores. Surface-tension effects between the differently oriented subgrains were often high enough to produce angular grain boundaries. Such irregular boundaries exhibited less tendency toward shearing than smooth regularly contoured boundaries.

Fig. 1—Shear along a grain boundary in high purity Al. Creep stress = 250 psi, ϵ_t = 0.07, T = 610°K. X100. Area reduced approximately 75 pct for reproduction.

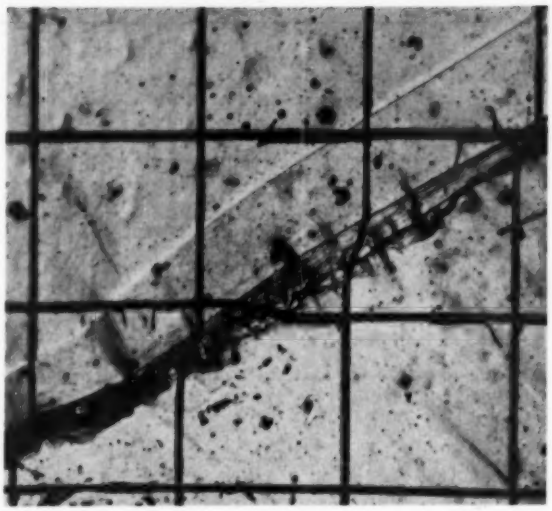

Fig. 2—Apparent bending of grid lines near the grain boundary as a result of alternate grain boundary shearing and migration in high purity Al. Creep stress = 250 psi, ϵ_t = 0.07, T = 747°K. X100. Area reduced approximately 75 pct for reproduction.

In view of the wide scatter in the individual shear displacements, k, an attempt was made to determine an average value $\bar{k}$. This was done by measuring each k value along five consecutive transverse grid lines, then skipping the next ten lines and repeating the measurement for the next five transverse grid lines, etc., until the entire gage section had been covered. In this way an average $\bar{k}$ was obtained over not less than 150 individual measurements per specimen. The longitudinal strain arising from grain boundary shearing then is readily determined by multiplying longitudinal components of the average shear displacement per grain boundary, $\bar{k}$, by the average number, n, of grain boundaries intercepted

Table II. Experimental Results

Temperature, °K	Time, Hr	ϵ_t	$\bar{k}$, In.	ϵ_{sb}	$\frac{\epsilon_{sb}}{\epsilon_t}$	ΔH, Cal per Mol
610	9.7	0.037	0.00035	0.0035	0.095	38,500
747	0.029	0.037	0.00031	0.0031	0.085	
610	20.4	0.070	0.00055	0.0055	0.079	35,000
747	0.105	0.070	0.00051	0.0051	0.073	
610	55.5	0.110	0.00085	0.0085	0.076	
640	14.42	0.110	0.00071	0.0071	0.065	38,000
700	1.215	0.110	0.00071	0.0071	0.065	
747	0.15	0.110	0.00075	0.0075	0.068	

per inch along the axis of the specimen, namely, $n = 10$. These data are incorporated in Table II.

Discussion of Results

Previous investigations[2-5] have established the validity of Eq. 2, and have shown that the activation energy for creep of pure aluminum and its dilute alloys is about $\Delta H = 36{,}000$ cal per mol independent of subgrain structures, cold work, or grain size. According to Eq. 2, the times to achieve the same strain for two creep tests under the same stress and different temperatures are given by

$$t_1 e^{-\Delta H/RT_1} = t_2 e^{-\Delta H/RT_2}. \qquad [3]$$

Using the times t and temperatures T given in Table II to achieve the same total strain, ϵ_t, the activation energies ΔH given in the last column of Table II were calculated. The agreement that was obtained with previous results again confirms the nominal validity of Eq. 2.

The data of Table II further reveal that essentially the same value of ϵ_{gb} is obtained for the same total creep strain, ϵ_t, independent of the test temperature. Consequently, these data suggest that

$$\epsilon_{gb} = f_{gb}\,(te^{-\Delta H_{gb}/RT}),\ \sigma = \text{constant} \qquad [4]$$

where the activation energy ΔH_{gb} for grain boundary shearing is identical with that for the total creep process, namely, about 37,000 cal per mol. These correlations suggest that the rate-controlling process for grain boundary shearing is the same as that for creep as a whole, since both processes exhibit the same activation energy. Since the activation energy for creep and stress-rupture[6] appears to be that for self-diffusion,[8] grain boundary shearing also appears to be dependent on a self-diffusion mechanism.

Rhines and Cochardt[10] investigated the shearing of bicrystals of high purity aluminum along their mutual grain boundary over the range from 473° to 923°K. They observed that the amount of grain boundary shearing increased with increase in difference of grain orientations. In another phase of their investigation they studied the effect of temperature on the displacement of the grain boundary under a stress of 100 psi for bicrystals whose relative crystallographic orientations were maintained constant for all samples tested. In terms of the suggestions of the current investigation, their results should be correlatable by means of the θ parameter. Such a correlation is shown in Fig. 5, wherein an average activation energy of about 40,000 cal per mol was obtained. Considering the difficulties in experimental techniques and the effect of sampling errors on this interesting investigation, the displacement θ curve appears excellent. The value of the activation energy obtained from Rhines and Cochardt's data is essentially equal to the activation energy obtained in the present investigation, and, furthermore, their results again suggest that the ΔH_{gb} for grain boundary shearing is practically identical with that for creep.

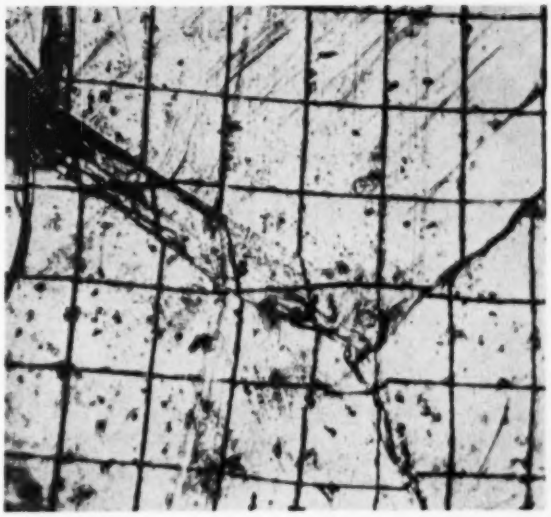

Fig. 4—Complex deformation of the grid near a grain boundary region due to multiple grain boundary shearing and migration processes. Creep stress = 250 psi, $\epsilon_t = 0.11$, T = 700°K. X50. Area reduced approximately 75 pct for reproduction.

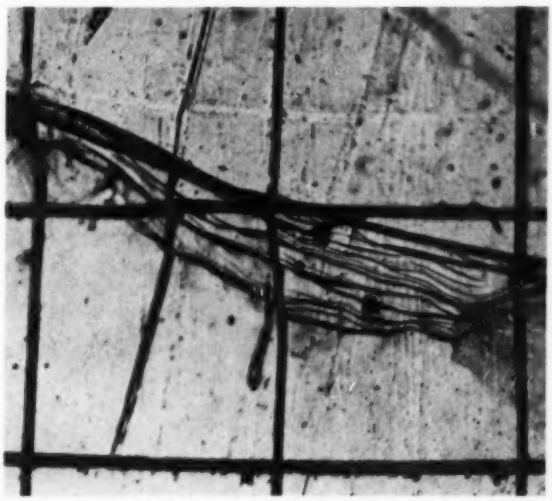

Fig. 3—Multiple boundary shearing and migration in high purity Al. Creep stress = 250 psi, $\epsilon_t = 0.07$, T = 747°K. X150. Area reduced approximately 75 pct for reproduction.

McLean[1, 4] has shown that the contribution of grain boundary shearing to the creep strain, ϵ_{gb}, varies with the duration of the test, t, in the same way as the total creep strain, ϵ_t, varies with time. The fact that the ratio of ϵ_{gb}/ϵ_t given in Table II is practically constant further substantiates McLean's original observations. Consequently, grain boundary shearing exhibits primary and secondary creep characteristics that are wholly compatible with those exhibited by the total creep strain. This is suggested also by the data, Fig. 5, deduced from Rhines and Cochardt's investigations. Obviously, the decreasing rate of

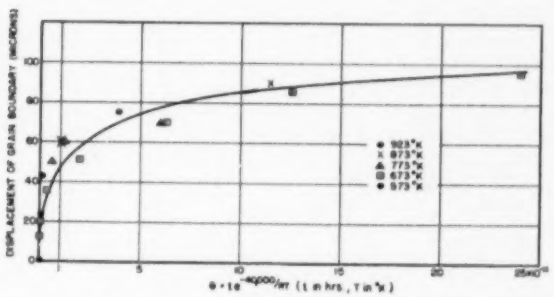

Fig. 5—Grain boundary displacement as a function of θ for high purity Al under a stress of 100 psi. Data of Rhines and Cochardt.[10]

grain boundary shearing during the primary stage of creep demands that structural changes are improving the resistance to grain boundary shearing in the same way as they serve to improve the total creep resistance. This strongly suggests that grain boundary shearing is dependent on the crystallographic processes of dislocation migration in the adjacent grains on each side of the boundary. This conclusion is substantiated further by the observation that polygonization is frequently more extensive in the vicinity of a boundary than in the core of the grains.

Perhaps grain boundary shearing occurs by the following mechanism: Shear stresses are relaxed at the grain boundary. Consequently, high bending and shear stresses are concentrated on the interpenetrating projections of each pair of grains across their mutual irregular boundary. If the stresses are relatively small, as in the case of damping capacity or shear modulus relaxation experiments, the straining occurs by shear displacements in the true grain boundary, and no permanent changes occur in the structure of the grains themselves. Under these conditions the properties of the grain boundary will be anelastic, exhibiting a viscous behavior. But under high stresses grain boundary relaxations induce sufficiently high bending and shear stresses on the grain projections in the vicinity of the grain boundary to cause these regions to undertake crystallographic mechanisms of deformation. Because of this bending, it might be expected that high polygonization would be induced in the grain boundary region as is observed experimentally. Furthermore, the accompanying strain as revealed by grain boundary shearing, ϵ_{gb}, should then follow the usual history-dependent laws for crystallographic mechanisms of deformation. The grain boundary itself merely serves to facilitate this crystallographic-deformation process by permitting concentrations of stress on the grain projections due to stress relaxations along the true boundary.

A large number of different investigators[10-12] have reported that the contribution of grain boundary shearing to the total creep strain increases with increasing temperature. But the validity of Eq. 4 and the experimental data recorded in Table II indicate that the contribution of grain boundary shearing to the total creep strain is independent of the test temperature. In many of the previous investigations, the higher temperature creep tests were conducted at lower stresses. Consequently, the increased contribution of grain boundary shearing to the total creep strain might have been ascribed to either the increased temperature of test or the lower stress. The present investigation suggests that temperature as such may not have been the factor responsible for the previous observations. And McLean's investigations[4] have shown that the relative contribution of grain boundary shearing to the total creep strain increases as the stress is decreased in a series of constant temperature creep tests. Consequently, the previously reported apparent increase of grain boundary shearing with increase in the test temperature might have been due to the simultaneous decrease in the applied stress.

Conclusions

1—The contribution of grain boundary shearing to the creep strain for high purity aluminum follows the functional relationship

$$\epsilon_{gb} = f(\theta),\ \sigma = \text{constant}$$

where σ is the creep stress, $\theta = te^{-\Delta H_{gb}/RT}$, t is duration of test, T is the test temperature in degrees absolute, R is the gas constant, and ΔH_{gb} is the activation energy for grain boundary shearing.

2—The activation energy for grain boundary shearing is about the same as that for creep. The contribution of grain boundary shearing to the creep strain follows the same primary and secondary stages as exhibited by the total creep strain. Extensive polygonization is observed in the vicinity of grain boundaries. These observations suggest that grain boundary shearing occurs by crystallographic mechanisms of deformation arising from bending and shearing of grain projections in the vicinity of the true relaxed grain boundary.

3—The relative contribution of grain boundary shearing to the total creep strain is independent of the test temperature, but as shown by McLean,[1-4] it increases as the stress decreases.

Acknowledgments

The authors wish to thank Mr. John Nadeau who contributed much to the initial phases of this work. In addition, they wish to express their appreciation to the Aluminum Co. of America for supplying the high purity aluminum used.

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aime NEWS

Bequests to AIME

Occasionally the Secretary has been asked how bequests may be made to the Institute. Unless the donor has some special use for the money in mind it is desirable to avoid specifying limitations and allow the Board of Directors to decide in what way the principal or income can best be used. If a specific purpose is expressed in the behest, it may develop that it is impractical to use the funds in the manner stated.

The following wording is suitable:

"I hereby bequeath to the American Institute of Mining and Metallurgical Engineers, Incorporated, a corporation organized under the Membership Corporations law of the State of New York, the sum of ———— to be used for the general purposes of the American Institute of Mining and Metallurgical Engineers at the discretion of its Board of Directors."

Utah Scene of Meeting

The AIME Rocky Mountain Region Industrial Minerals Conference will be held Oct. 28 to 30, 1954 in Salt Lake City, Utah. Headquarters for the meeting will be the Newhouse Hotel, and registration will start Thursday, October 28. Numerous papers of interest to Metals Branch members will be presented.

The morning session on Friday, October 29, with R. C. Talbott, Columbia-Geneva Steel Div., and R. R. Williams, Jr., Colorado Fuel & Iron Corp., as Co-Chairmen includes the following papers: *Industrial Water* by ElRoy Nelson, First Security Corp.; *The Role of Industrial Minerals in Utah's Steel Industry* by John K. Hayes, Columbia-Geneva Steel Div.; *Natural Gas in the Intermountain Area* by H. F. Hillard, Mountain Fuel Supply Co.; and *Chemical Treatment of Ores* by C. A. Romano, Intermountain Chemical Co. The afternoon session includes the paper *Titanium* by C. C. Blalock, Colorado Fuel & Iron Corp.

The Industrial Minerals Luncheon to be held on Friday will have as featured speaker the Governor of Utah, J. Bracken Lee. Arrangements have been made for members to attend the Utah-Idaho football game Saturday afternoon. Buses will transport those attending the game to and from the Newhouse Hotel.

The meeting will close with the Utah Section AIME Annual Fall Cocktail Party and Dinner Dance in the Grand Ballroom of the Newhouse Hotel. The date of the dance is Saturday, October 30, at 7:00 pm.

NOHC Buffalo Section Holds First Golf Party

The Buffalo Section, NOHC, held its first annual golf party at the Lancaster Country Club, Lancaster, N. Y. on May 19, 1954. The suppliers Committee headed by G. W. Edwards, Basic Refractories, Inc., and George A. Brunner, E. J. Lavino Co., provided golf prizes, door prizes, and a cocktail party. A total of 115 registered for the event with 64 persons playing golf.

Anyone wishing to be informed of the annual meetings and golf parties should write to Harry A. Morlock, District Sales Manager, General Refractories Co., 807 Genesee Bldg., Buffalo 2, N. Y., who is Secretary of the Buffalo Section.

Schedule Final For International Meeting

Plans have been completed for three international engineering meetings to be held in Rio de Janeiro and São Paulo, Brazil, between July 25 and Aug. 12, 1954.

The Sectional Meeting of the World Power Conference is scheduled for Rio de Janeiro, July 25 to August 10. The Fourth Convention of Inter-American Assn. of Sanitary Engineering is slated for São Paulo, July 25 to 31. The Third Convention of Pan American Federation of Engineering Societies (UPADI) is planned for São Paulo, August 2 to 12.

ASCE Raises Dues to $25

By a vote of 72.7 pct of the approximately 16,000 members voting, the dues of Members, Associate Members, and Affiliate Members of the American Society of Civil Engineers have been raised from $20 to $25. Junior Member dues have been raised from $10 to $15.

Coming Events

July 19-20, International Conference, Mechanical Effects of Dislocations in Crystals, University of Birmingham, England.

July 21-28, International Union of Crystallography, general assembly and international congress, Paris.

July 25-Aug. 10, World Power Conference, sectional meeting, Rio de Janeiro, Brazil.

Aug. 2-12, Pan American Federation of Engineering Societies (UPADI), 3rd convention, Sao Paulo, Brazil.

Aug. 23-27, Oak Ridge Summer Symposium, Modern Analytical Chemistry, Oak Ridge, Tenn.

Sept. 8-10, ASME, fall meeting, Schroeder Hotel, Milwaukee.

Sept. 12-16, American Institute of Chemical Engineers, Hotel Colorado, Glenwood Springs, Colo.

Sept. 14-24, Instrument Society of America, Philadelphia.

Sept. 20-24, American Mining Congress, Civic Auditorium, San Francisco.

Sept. 24, AIME, MBD, fall meeting, Fairmont Hotel, San Francisco.

Sept. 28-Oct. 1, Assn. of Iron & Steel Engineers, iron and steel exposition, Cleveland Public Auditorium, Cleveland.

Sept. 30, AIME, Utah Local Section. Stag. Cocktails, dinner, smoker, Newhouse Hotel.

Oct. 1-2, Standards Engineers Society, annual meeting, Haddon Hall, Atlantic City, N. J.

Oct. 3-7, Electrochemical Society, Inc., Statler Hotel, Boston.

Oct. 18-22, National Safety Congress and Exposition, Chicago, Ill.

Oct. 18, AIME, Columbia Local Section, Spokane, Wash.

Oct. 26, Assn. of Consulting Chemists and Chemical Engineers, Inc., annual symposium and banquet, Hotel Belmont Plaza, New York.

Oct. 29, AIME, NOHC and Pittsburgh Local Section, off-the-record meeting, William Penn Hotel, Pittsburgh.

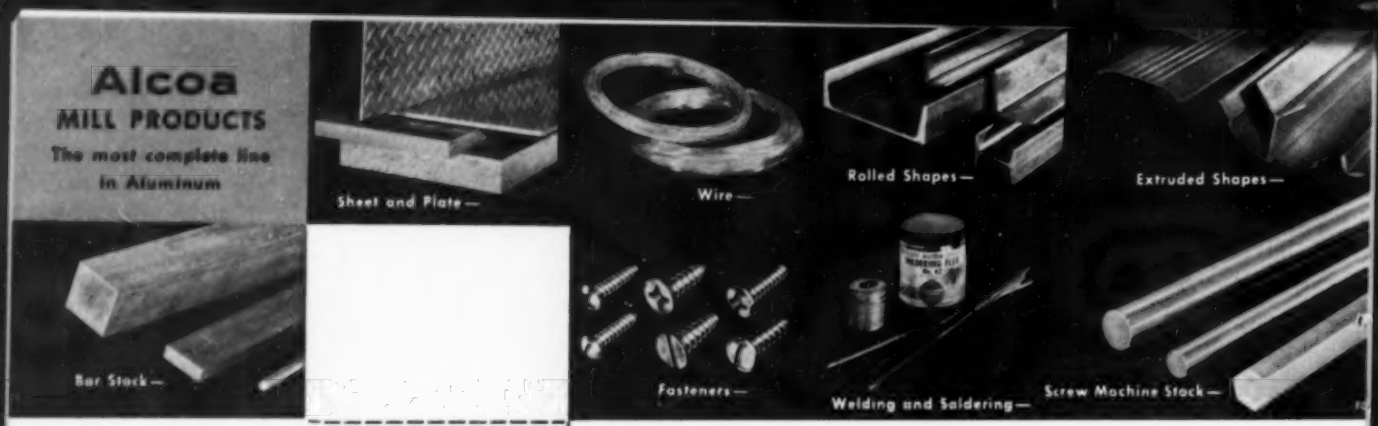
Nov. 1-3, AIME, Institute of Metals Div., fall meeting, Hotel Morrison, Chicago.

Nov. 1-5, American Welding Society, Hotel Sherman, Chicago.

Nov. 1-5, American Society for Metals, National Metal Congress, Palmer House, exposition, International Amphitheatre, Chicago.

Dec. 1-4, AIME, Electric Furnace Steel Conference, William Penn Hotel, Pittsburgh.

Dec. 12-15, American Institute of Chemical Engineers, annual meeting, Statler Hotel, New York.



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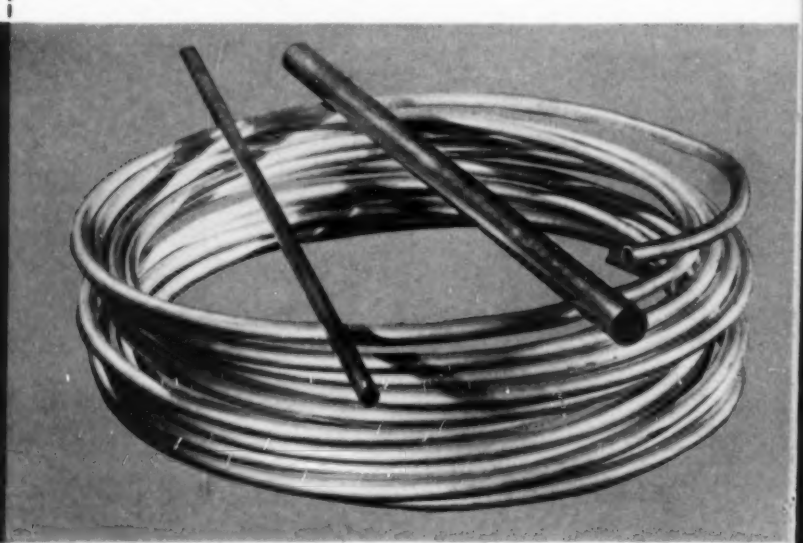

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Personals

J. A. MacMillan has joined the Globe Iron Co., Jackson, Ohio, as general superintendent. Mr. MacMillan had been assistant general superintendent of the Jackson Iron & Steel Co.

J. T. Shimmin has accepted a position with North American Aviation, Inc., Downey, Calif., as an engineer-research. He had been associated with the Titanium Metals Corp. of America, Henderson, Nev., as shift foreman.

H. A. White, Smeeth-Harwood Co., Chicago, was recently elected president of the Non-Ferrous Founders' Society. Mr. White has served as first vice-president during the past year. Mr. White is also General Chairman for the AIME Annual Meeting to be held at Chicago in 1955. Mr. White has purchased the entire interest of John M. Dixon and continues as sole proprietor of Smeeth-Harwood Co.

Richard C. Cole has resigned as ore buyer for the American Smelting & Refining Co., New York. He is now plant manager with Vitro Uranium Co., Salt Lake City, Utah.

T. J. McLOUGHLIN

Thomas J. McLoughlin, formerly assistant to vice president of operations-steel, U. S. Steel Corp., has become vice-president for the Blocked Iron Corp., Albany, N. Y. Mr. McLoughlin has been a Director of AIME and also was Chairman of the Iron and Steel Div.

A. S. Cogan, Olin Industries, Inc., has been transferred from the East Alton, Ill., plant to the Winchester Repeating Arms Div., at New Haven, Conn.

Arvid N. Anderson has joined the Cleveland foundry of the Aluminum Co. of America as junior metallurgist. He recently graduated from the Colorado School of Mines.

A. B. KINZEL

Augustus B. Kinzel has been appointed director of research, Union Carbide & Carbon Corp. Dr. Kinzel will be responsible for the administration and coordination of the research activities of all divisions of UCC. He has been actively engaged in research work with UCC since 1926, when he joined the Electro Metallurgical Co. as a research metallurgist. Dr. Kinzel became chief metallurgist of the laboratories in 1931 and a vice-president of Electro Metallurgical Co. in 1944. More than 40 U. S. patents have been issued to him. He pioneered in the theory of stainless steels and the development of weldable alloy structural steels. He has contributed to the research and development achievements of high vanadium high speed steels; vanadium, chromium, and manganese metals; and extra low carbon ferrochrome alloys. Electromet's new process for titanium crystals is largely creditable to Dr. Kinzel's research leadership. He is a Vice-President of AIME, and holds membership in the ASM, ASME, ASTM, AWS, and International Acetylene Assn. He is chairman of the Welding Research Council, vice president of the International Institute of Welding, and chairman of the advisory committee to the Institute of Mathematical Sciences, New York University. He is presently chairman of the Naval Research Advisory Committee and chief consultant in metallurgy to Los Alamos Laboratories and other units of the Atomic Energy Committee.

A. C. Burningham has resigned as joint managing director of the Eaglescliffe Chemical Co., Ltd., although he remains on the board. He is with British Chrome & Chemicals Ltd., as deputy managing director.

Edgar W. Engle, manager of product and process development engineering, Carboloy Dept., General Electric Co., Detroit, has been named manager of cemented carbide products engineering. Mr. Engle has been with Carboloy since 1941.

W. Bryan McPherson is now employed at the Sivyer Steel Casting Co., Milwaukee.

L. S. Piper recently was made superintendent of engineering services, engineering div., Consolidated Mining & Smelting Co. of Canada Ltd., Trail, B. C. Mr. Piper had been acting smelter superintendent, smelting dept. He has been with Cominco since 1928.

Lee H. Coleman was recently honored by the Leeds & Northrup Co., Philadelphia, on the 25th anniversary of his employment by the company. He joined the firm in 1929 as a sales engineer at Chicago. In 1951 he became head of the steel and ceramics section, market development dept. In May 1954 Mr. Coleman was transferred to the Philadelphia office.

S. H. BUSH

Spencer H. Bush is in charge of physical metallurgy research with the General Electric Co. applied research subsection, engineering dept., at the Hanford atomic installation near Richland, Wash. Dr. Bush was formerly a metallurgist in applied research subsection assigned to pile metallurgy problems with General Electric at Hanford.

R. S. McLaren was promoted to assistant director of research, Shawinigan Water & Power Research Dept., Shawinigan Falls, Que., Canada.

F. H. Atwood, vice-president of Harbison-Walker Refractories Co., Pittsburgh, was reelected president of the Refractories Institute, Pittsburgh.

Proposed for Membership

— Metals Branch AIME —

Total AIME membership on June 30, 1954 was 21,033; in addition 1697 Student Associates were enrolled.

ADMISSIONS COMMITTEE

O. B. J. Fraser, Chairman; R. B. Caples, Vice-Chairman; V. A. Ayer, A. C. Brinker, R. H. Dickson, Max Gensamer, Ivan A. Given, Fred W. Hanson, T. D. Jones, Sidney Rolle, J. H. Scaff, John T. Sherman, F. T. Sisco, Frank T. Weems, R. L. Ziegfeld.

The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

In the following list C/S means change of status; R, reinstatement; M, Member; J, Junior Member; A, Associate Member; S, Student Associate.

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Munster—Sullivan, James P. (A)
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Detroit—Spooner, Norman F. (M) (R. C/S—S-M)
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Houston—Trubey, George A. W. (M)

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Toronto, Ont.—Morrison, Bernard H. (M)

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Letchworth, Herts—Brearley, Alan C. (M)

Necrology

Date Elected	Name	Date of Death
1898	James Colquhoun	June 17, 1954
1917	Walter James Eaton	June 5, 1954
1917	W. O. Hotchkiss	June 20, 1954
1951	Otto Henry Metzger	Apr. 17, 1954
1909	Stephen Royce	June 12, 1954
1949	Charles H. Scheuer	Unknown
1946	K. A. Spohr	May 9, 1954
1937	G. T. Walters	May 26, 1954

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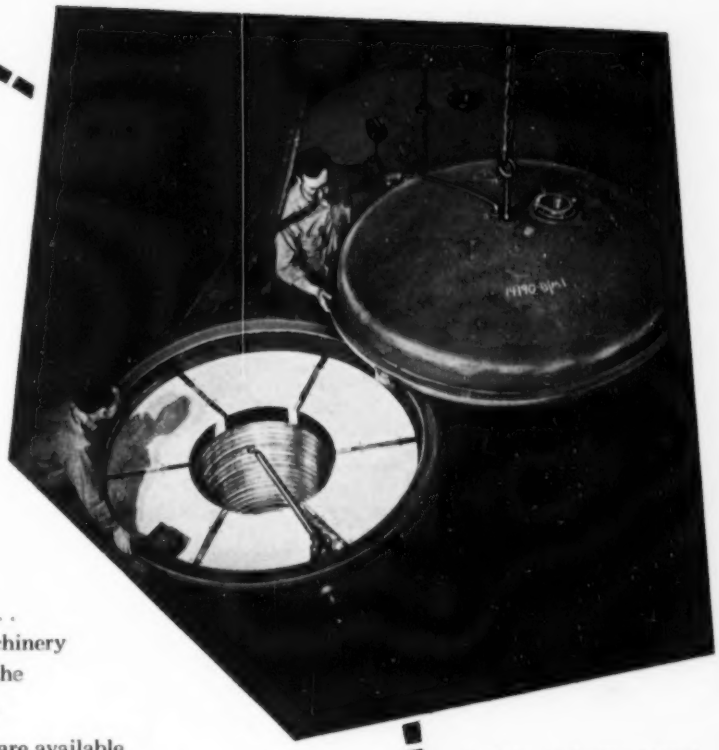
* Previous issue.

It's **Experience** *that Counts in Building Vacuum Furnaces*



A Stokes induction-heated vacuum melting furnace.

Fifty years of working with vacuum techniques . . . fifty years of building *practical* vacuum equipment . . . plus sixty years of making precision machinery . . . are Stokes' unique background for the production of efficient vacuum furnaces.

Stokes vacuum furnaces of many types are available for melting and casting gas-free metals of extreme purity, high ductility and great strength; for sintering, annealing and degassing. Designs include furnaces for top or bottom pouring, for simple or multiple ingots, for centrifugal casting. We build tilting type induction-heated melting furnaces of 10 to 1000 pounds capacity; movable retort resistance-heated furnaces for degassing and annealing in 10, 20 and 30-inch retort sizes; two-zone furnaces with movable boat; and others.

Stokes Vacuum Furnace designs provide for future as well as for immediate needs. Ports for extra pumps, leads for extra heat input, room for larger crucibles, etc., are engineered into original equipment so that growing requirements and changing conditions can be met at minimum cost.

Write for the latest information on Stokes Vacuum Furnaces. Above all, visit the Stokes plant at Philadelphia and see at first hand the finest engineering and manufacturing facilities in the vacuum furnace manufacturing field.

F. J. Stokes Machine Company
Philadelphia 20, Pa.

Operator regulates booster pump from central control panel. This is one of a battery of 16 Stokes Vacuum Furnaces in a major installation.

STOKES

STOKES MAKES: High Vacuum Equipment, Vacuum Pumps and Gages / Industrial Tabletting, Powder Metal and Plastics Molding Presses / Pharmaceutical Equipment

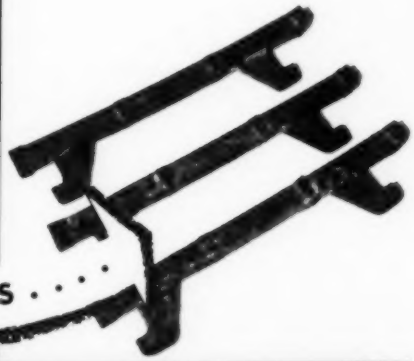
YOUR SINTERING MACHINE Costs may be CUT ...by USING GRATE BARS of 77-NI

1,408 Grate Bars of 77-NI (special alloyed steel) produced by CARONDELET are still serving in the sintering machine shown above in the plant of the Blackwell Zinc Co., Inc., of Blackwell, Okla. This machine has an effective grate area 12 ft. wide and 168 ft. long.

...A HEAT AND ABRASION RESISTANT SPECIAL ALLOYED STEEL GIVING LONGER SERVICE LIFE

PRODUCED BY CARONDELET

Asked to cast Grate Bars that would out-perform ordinary gray or malleable irons in overcoming burning, warpage and abrasion, CARONDELET produced those shown herein of 77-NI using CARONDELET'S Electric Furnace process for metallurgical control.



STILL IN SERVICE AFTER 2½ YEARS....

Still in use after 2½ years, the above Grate Bars represent an extended service life that promises to reach approximately five years! The ratio of their service life to usual grate bar materials ranges from 6 to 1 to 10 to 1 as proved by this demonstrated service and others in non-ferrous sintering.

SPECIAL GRAY IRON GRATE BARS LASTED LESS THAN 6 MONTHS...

Alloyed heat-resistant gray iron Grate Bars used concurrently to test a modified design, lasted less than 6 Months.

SEND FOR DESCRIPTIVE BOOKLET ...THAT PRESENTS IN DETAIL THE OPERATION AT CARONDELET TO THE BENEFIT OF VARIED INDUSTRY

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